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# QUALITY ASSURANCE MANUAL
# ACIDIC PRECIPITATION IN ONTARIO STUDY (APIOS)
# DEPOSITION MONITORING NETWORKS

ARB-051-85-AQM
APIOS-006-85
FEBRUARY 1985

Prepared for

Ministry of the Environment
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario

By

Concord Scientific Corporation
2 Tippett Road
Downsview, Ontario
M3H 2V2

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# 1. INTRODUCTION

The Air Resources Branch of the Ontario Ministry of the Environment currently operates two networks under the auspices of the Acidic Precipitation in Ontario Study (APIOS). Both networks are designed with the general objective of quantifying wet and dry deposition of acid related pollutants across the Province of Ontario. These networks are the APIOS Event Wet/Dry Deposition Network and the APIOS Cumulative Wet/Dry Deposition Network. The event network collects precipitation and air filter samples on a daily basis for the determination of both wet and dry short term deposition and to determine the origin of the pollutants deposited in these events; the cumulative network collects samples on a 28-day cycle (monthly basis since the network inception to December, 1981) for the determination of long-term wet deposition patterns and the estimation of dry deposition.

Further details of the objectives of each network can be found in the Technical and Operating Manual (Bardswick, 1984).

The APIOS air and precipitation monitoring program has been in operation for several years, and is currently using well documented operating procedures which have been implemented and revised for all aspects of the network operation. A detailed quality assurance plan has also been written to describe the network's quality assurance objectives

for all aspects of the program as well as to provide a guide to the general QA/QC procedures that are to be employed in the network operations.

This QA Manual will serve as a QA reference for all personnel within the program by documenting the specific QA/QC procedures, requirements, schedules, reporting mechanisms, etc. that should be followed for all aspects of the program.

This manual is designed to complement, not replace the network operations manual or QA plan (Bardswick, 1984), and should be used as a reference document for specific QA/QC requirements and procedures for each aspect of the network operations. The document control system followed is the same one used in other APIOS documentation and therefore may be easily updated or modified as network QA/QC requirements change.

The manual addresses the organizational structure in Chapter 2. Chapter 3 gives a description of the general network QA/QC procedures and requirements that apply to all areas of the network. Chapters 4 and 5 discuss the field and laboratory QA/QC requirements respectively, while Chapter 6 discusses data related QA/QC.

## 2. ORGANIZATIONAL STRUCTURE

An organizational chart is shown in Figure 2.1, and displays staff relationships, lines of authority, and responsibilities. The deposition studies are undertaken by the Special Studies Unit which is part of the Atmospheric Research and Special Programs Section of the Air Resources Branch. Each position in the organizational chart is listed below along with a brief description of responsibilities and duties as they relate to the APIOS monitoring program.

Atmospheric Research and Special Programs Supervisor:

- oversees management of Special Studies Unit's programs.

Special Studies Unit Head:

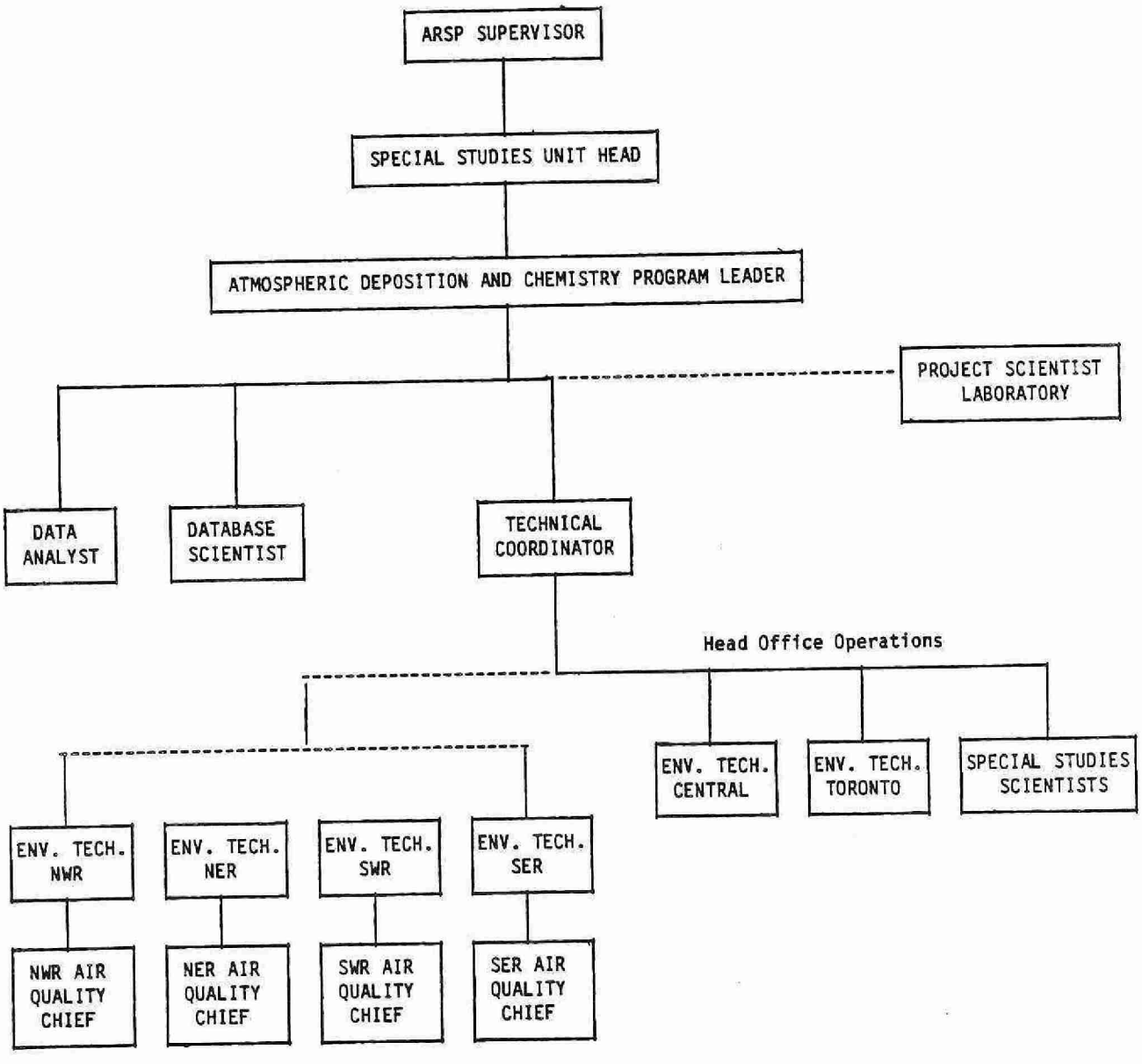
- program planning, supervision and review,
- budget approval and review,
- external program representation,
- scientific program guidance within Branch,
- public relations as required.

Atmospheric Deposition and Chemistry Program Leader (Network Manager):

- scientific direction and supervision of program staff,
- overall responsibility for all aspects of atmospheric deposition and chemistry program,

FIGURE 2.1

Organizational Chart



- program and budget planning,
- responsibility for program report preparation,
- maintain scientific profile of program outside of the Branch,
- quality assurance manager.

Data Analyst:

- advanced data analysis,
- interpretive report preparation,
- scientific information exchange.

Database Scientist:

- database development and maintenance,
- data validation and quality control,
- production of data reports, statistical data summaries,
- supervision of interfacing and data exchange with other networks,
- production of data related aspects of QA reports.

Technical Coordinator (Quality Assurance Coordinator):

- oversee regional network operations,
- supervision of special studies in the areas of instrument development, evaluation, and intercomparison,
- quality assurance coordinator,
- preparation of quality assurance reports,
- maintenance of document distribution list.

Project Scientist - (Laboratory):

- liaison between laboratory operations and monitoring program,
- supervision of laboratory operations,
- preparation of laboratory reports,
- participation in external laboratory QA/QC activities.

Special Studies Scientist:

- conduct and prepare reports of all special studies,
- support of routine and special study quality assurance,
- support of network operations.

Environmental Technicians (Regions):

- installation, operation and maintenance of monitoring network sites,
- liaison (supervision, training, evaluation, etc.) with network operators,
- execution of special studies as required.

Environmental Technician (Toronto):

- sample coordinator, submission of all samples,
- filter preparations,
- inventory maintenance and technical field support.

Note: The environmental technicians in the regional offices are supervised by regional Senior Technicians or Air Quality Chiefs with co-ordination of APIOS activities from APIOS head office.

It is the responsibility of the quality assurance coordinator to ensure that the field personnel are meeting their APIOS field requirements by designing and implementing the QA/QC system required to monitor field QA/QC procedures. In addition, a sound system for co-ordinating APIOS activities over all regions is to be employed. Regional managers (air quality chiefs) are to be involved in the relevant aspects of field program planning, and a mechanism is to be implemented for incorporating their input into the network operating system. To accomplish this, regional air quality chiefs and APIOS network managers are to meet on an annual basis to discuss these aspects of the program (see Section 3.8, Annual Network Review).

# 3. GENERAL NETWORK QA/QC PROCEDURES AND REQUIREMENTS

This section discusses some of the important aspects of the network quality assurance and quality control system in general terms. The quality assurance objectives and the network approach for document control, sample custody, training, corrective action, audit requirements, special studies QA, and QA reporting are presented in this section, while the specifics for each of these QA elements are presented in the subsequent sections dealing with field, laboratory and data operations respectively.

## 3.1 Documentation and Document Control

Network documentation and document control provide the means for ensuring that all areas of the network are properly documented and that these documents are produced, distributed, reviewed and revised on a regular basis. A list of network documentation is summarized in Table 3.1.

A documentation control system is to provide the means for revising, updating and distributing network documents by 1) providing an indexing system to facilitate the insertion of revised information into an existing document and 2) maintaining a distribution list of personnel or organizations receiving all documents to ensure that all are made aware of changes as they occur. The section/page indexing system des-

TABLE 3.1

APIOS Network Documentation

| TITLE | CONTENTS |
|---|---|
| Technical and Operating Manual | - description of instrument and sample handling procedures for technicians and operators, routine QA/QC procedures, corrective action requirements, calibration and maintenance, data management, laboratory handling procedures |
| Network Instrumentation Manual | - description of instruments, trouble shooting, wiring schematics, etc. |
| Data Operations Manuals | - screening and validation procedures; data reporting formats for published data |
| Quality Assurance Manual | - specific QA procedures implemented from QA plan |
| Quality Assurance Plan | - network QA objectives and implementation plan |
| Network Descriptions | - overview of network mandates and objectives |

TABLE 3.1 (cont'd)

APIOS Network Documentation

| TITLE | CONTENTS |
|---|---|
| Site Documentation | - detailed description of all network sites for distribution to users as required |
| Site Operation Log Books | - summary of samples submitted, sampling and instrument problems |
| Instrument Logs | - summary of sampling history, problems, maintenance record, calibration factors, etc. |
| Data Logs | - summary of data editing screening and matching problems |
| Data Reports | - annual reports of quality assured data for all networks |
| Quality Assurance Reports | - result of network QA program, etc. and general assessments of network performance |

TABLE 3.1 (cont'd)

APIOS Network Documentation

| TITLE | CONTENTS |
|---|---|
| Audit Reports | - results of external audits |
| Data Transmittal Documentation | - description of network, data archive structure and raw data tapes distributed to external users |
| Sample Submission Forms | - field information and sample I.D., comment codes, etc. for field, laboratory and data centre |
| Site Description Questionnaires | - site location, description, operator information, etc. |
| Site Change Forms | - description of changes to sites |
| Corrective Action Form | - description of corrective action required and assignment of responsibility |
| QA/QC Sample Schedule | - schedule for preparation and submission of QA/QC samples |

cribed by the USEPA (1976) and adopted in other APIOS documentation has been followed in this manual.

In this system the following information is recorded in the upper-right corner of each page:

Section No.:

Revision No.:

Date of Revision:

Page ___ of ___

Each major section starts on a new page, and the total number of pages in the section is noted on each page as well as in the table of contents. As revisions occur, the pages of the old section are simply replaced with the revised version and the table of contents is corrected. This system avoids reproducing an entire document for the sake of revisions to part of it. The table of contents consists of section and subsection number and title, pages and date of revision.

As described in the QA plan, the network distribution list should have the names and addresses of all personnel receiving network documentation on file. It is the QA coordinator's responsibility to maintain this distribution list and to ensure that all documents and revisions are properly circulated. It is the responsibility of the

network technicians to ensure that all on site operating manuals are up to date.

## 3.2 Sample and Data Custody

Sample and data custody monitoring form the basis of the system used to track precipitation and air samples and their associated data forms through all aspects of network operations. It is an important aspect of overall network quality assurance because it provides the means of ensuring that all data collected and reported in the field are accurately and completely reported in the final database, and that proper sample shipping and storage procedures are followed in all areas of the program.

From a quality control point of view, the APIOS sample and data submission and tracking system has been designed to meet this general objective through the use of specific procedures. These include comprehensive operator, field and laboratory data submission sheets, the use of duplicate log books and data sheets for many stages of the sample submission procedures, unambiguous sample identification and labelling systems, well conceived and documented procedures for sample reception, shipment and storage in the field and at the laboratory, and a sophisticated computerized laboratory sample tracking system (LIS) to assign, schedule and report analytical results within suitable time periods. However, from a quality assurance point of view, the network must ensure

that proper checks are applied to monitor the effectiveness of these systems.

The specific sample and data custody QA/QC requirements are discussed for each sample type in Sections 5 through 7 below. However, there are some general procedures and concepts that apply to all samples that will be discussed here.

The general procedures for sample processing in this program are:

1) the site operator completes operator sheets summarizing sample data, sample type (i.e. routine sample or QC sample, etc.), instrument operation, and potential contamination and submits them along with the samples to the regional APIOS technician;

2) regional technicians then assign field sample I.D. numbers to the samples (using the duplicate label system to avoid errors), check the information on the operator sheets, enter results of field measurements (e.g. pH, volume, for event samples etc.) and calculations (e.g. gauge depth) and transfer all relevant information to the field submission sheet and submit samples to the laboratory for analysis, data sheets to the head office for processing and sample summaries to the QA Coordinator;

3) samples are then received at the laboratory reception area, where they are processed by the head office environmental technician who:

- checks all samples,
- prepares laboratory submission forms identifying analyses required,
- sends shipping containers back to the field,
- initiates laboratory data/sample processing through LIS;

4) field data are received at head office and processed to create computer data files for subsequent merging with laboratory analysis results to form raw data files;

5) raw data files are reviewed, validated and screened before creating final data sets.

In carrying out general sample submission procedures, there are a number of important QA/QC steps that are to be carried out to ensure that complete and accurate data are transferred through different components of the system. These are described below.

1) Sample Logs - are to be used at critical sample transfer points to ensure that all samples and data received are passed on to the next stage. The important stages to be monitored include the operator/technician transfer, technician/sample

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reception transfer and laboratory/head office transfer. The purpose of these logs is to ensure that all samples and data are passed on, and that any errors in transmittal are detected.

2) Technicians are to keep duplicates of all field sheets submitted and routinely compare them to operator sheets to ensure all data have been properly processed before shipping samples to the laboratory.

3) The technician network summary sheet system is to be employed to summarize event network submissions (Figure 3.1) on a monthly basis. A similar summary sheet for the cumulative network is presented in Figure 3.2.

4) The technician summary sheet information is to be carefully reviewed by the QA Coordinator on a continuous basis to ensure that the proper QA/QC procedures are being applied.

5) Access to the network database is to be restricted to ensure that a limited number of personnel are permitted to make changes to the database, and all such changes must be authorized by the database scientist.

FIGURE 3.1

Event Network Summary Sheet

**Collection of Event Samples**



Region: _____

Month: _____

Year: _____

| Site | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Day | Vol. | G.D. | Vol. | G.D. | Vol. | G.D. | Vol. | G.D. | Vol. | G.D. | Vol. | G.D. | Vol. | G.D. |
| 1 | | | | | | | | | | | | | | |
| 2 | | | | | | | | | | | | | | |
| 3 | | | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | | | |
| 10 | | | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | | | |
| 13 | | | | | | | | | | | | | | |
| 14 | | | | | | | | | | | | | | |
| 15 | | | | | | | | | | | | | | |
| 16 | | | | | | | | | | | | | | |
| 17 | | | | | | | | | | | | | | |
| 18 | | | | | | | | | | | | | | |
| 19 | | | | | | | | | | | | | | |
| 20 | | | | | | | | | | | | | | |
| 21 | | | | | | | | | | | | | | |
| 22 | | | | | | | | | | | | | | |
| 23 | | | | | | | | | | | | | | |
| 24 | | | | | | | | | | | | | | |
| 25 | | | | | | | | | | | | | | |
| 26 | | | | | | | | | | | | | | |
| 27 | | | | | | | | | | | | | | |
| 28 | | | | | | | | | | | | | | |
| 29 | | | | | | | | | | | | | | |
| 30 | | | | | | | | | | | | | | |
| 31 | | | | | | | | | | | | | | |

Codes: 0 - no volume collected but gauge depth taken
( ) - multiple day sample
— - no precipitation
S - spilled sample
T - trace sample
M - sampler malfunction
FB - field blank
D - duplicate
C - composite

Comments: _____

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FIGURE 3.2

APIOS Cumulative Network Summary Sheet

Region: _____

Technician: _____

Sampling End Date: _____

Sample Summary

| Station Name | Number | Instrument Malfunction | | Sampling Problems | | Specify Malfunction or Problem | Corrective Action Taken |
|---|---|---|---|---|---|---|---|
| | | Precip. | Air | Precip. | Air | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

Codes:

| | | | |
|---|---|---|---|
| Malfunction affected sample | 1 | Sampling problems affected sample | 1 |
| Malfunction did not affect sample | 0 | Sampling problems did not affect sample | 0 |
| No malfunctions | - | No sampling problems | - |

Note: Indicate if any composites were handled and provide the dates and times of the composite handling.



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## 3.3 Training and Upgrading

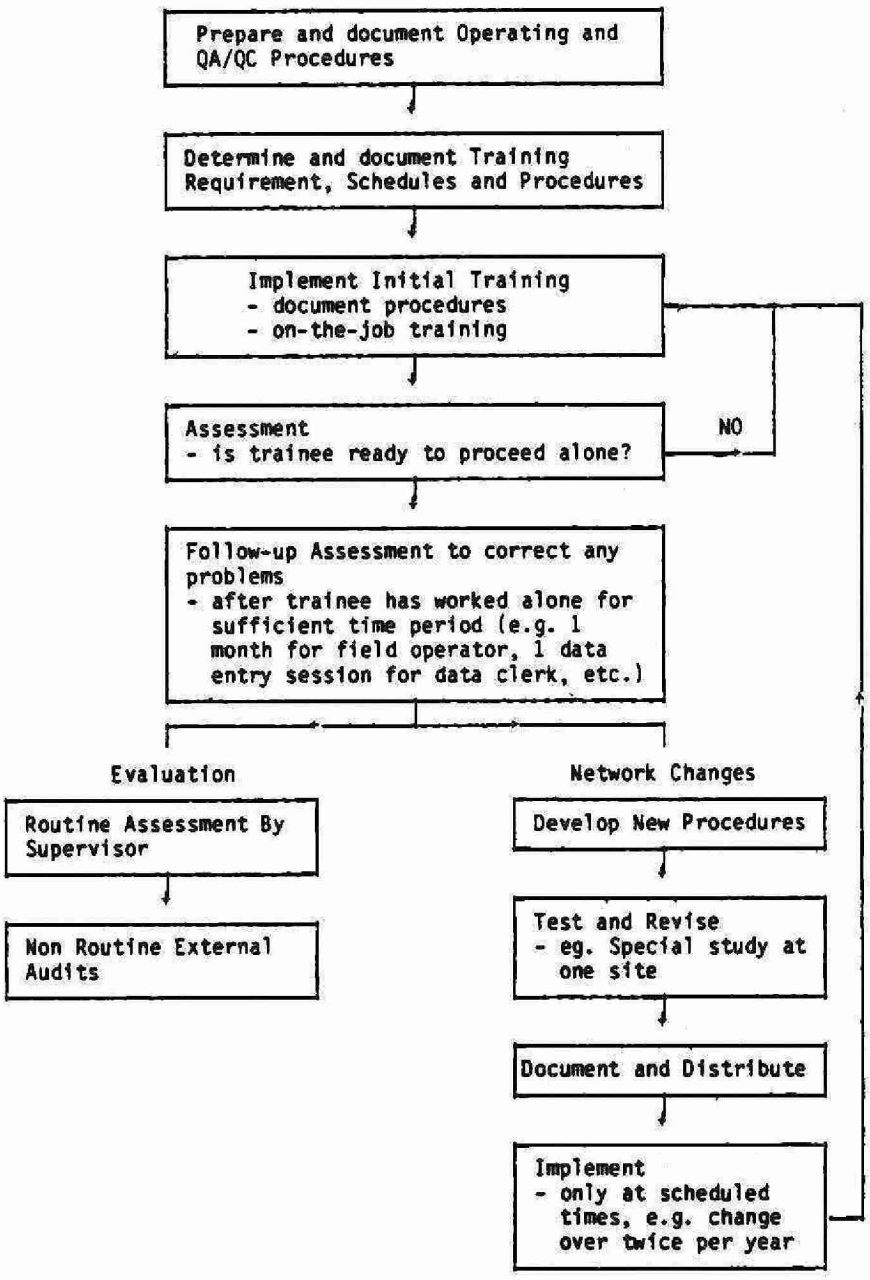
The general objectives of the network training and upgrading procedures are, to 1) ensure that all network personnel are aware of their duties and have been properly instructed in the execution of those duties at the beginning of their employment, 2) ensure that all personnel are informed and trained in the event of changes to procedures, 3) ensure that operator, technician, lab analyst and data personnel skills do not deteriorate with time, and 4) to provide the means of detecting problems or shortcomings in the network training system.

To meet the general training objectives, it is essential that the managers within each general area of the network (i.e. field operations, data management, laboratory and quality assurance) document a formal set of training requirements and procedures that are designed to meet the appropriate level of training for the respective positions. It is the QA Coordinator's responsibility to monitor the effectiveness of the various training programs. There are two major components to a comprehensive and effective training program; namely 1) a formal set of training, evaluation and follow up procedures and 2) effective communications (formal and informal) between various members of the program.

The important stages of a formalized training and upgrading plan are summarized in Figure 3.3 and are discussed below.

FIGURE 3.3

Summary of Formalized Training Plan



## Training Requirements and Schedules

Training requirements for new employees, and existing network staff are quite different. In general, new site operators are trained by the regional technicians who are, in turn, trained by the technical coordinator and other head office personnel. Laboratory analysts have a separate training program, while data clerks and data management personnel are trained by the database and network management personnel. The key steps in training new personnel in different aspects of the program are to consist of:

1) providing copies of all relevant network documentation;

2) having all duties described and explained by the immediate supervisor;

3) undergo on-the-job training with appropriate personnel for suitable time periods;

4) perform an assessment after the training period to ensure all procedures (after working alone for a short period) are properly followed and to avoid the development of "bad habits".

Training requirements for new employees consist mainly of educational and familiarization procedures. The specific training

requirements for new employees are presented in Table 3.2 for each network position.

Training and evaluation requirements for existing employees differ from those of new employees because they consist of 1) reinforcement of existing training as a result of performance evaluations or audits to maintain a high level of performance, and 2) training in new procedures introduced as a result of changes to the network. These types of ongoing training and evaluation requirements are particularly important for the field operators, regional technicians and laboratory analysts. The specific training and evaluation procedures that are to be applied on a routine and continuing basis are presented in Table 3.3.

## Assessment and Follow-Up

It is essential that the effectiveness of the network training program be assessed on a regular basis, and that any shortcomings in the performance of the training program or individual project members be corrected in a timely and efficient manner. A detailed evaluation of any new employee is to be carried out within three months of beginning employment as part of the internal auditing procedures. It should be the responsibility of the employee's immediate supervisor to carry out the evaluation (e.g. regional technician for operators, air quality chiefs for regional technicians etc.) and the results are to be reported to the quality assurance coordinator who is then responsible (as part of

TABLE 3.2

Summary of Training Requirements for New Employees

| Position | Training Requirements |
|---|---|
| Field Operator (Trained by regional technician) | 1. Network documentation including overviews and procedures.<br>2. Verbal explanation of network objectives.<br>3. Explanation of operating procedures and how to use them.<br>4. Step by step explanation of sample collector duties stressing importance of sample contamination.<br>5. Step by step illustration of all procedures by regional technician.<br>6. Example of completed data sheets and log book requirements.<br>7. Operator run through procedures with technician.<br>8. Explanation of QA/QC procedures and why they are important.<br>9. Illustration of instrument checks and how to "trouble shoot" common problems.<br>10. Corrective action procedures to follow when problems arise.<br>11. Follow up - regional technician to make a return visit as soon as possible to evaluate the adequacy of the operator training.<br>12. Regional technician to maintain close, informal contact with new operators to help solve and avoid problems.<br><br>Note: Frequency of technician visits with operators to be increased (1/week for event, 1/month for cumulative) during the first few months. |

TABLE 3.2 (cont'd)

Summary of Training Requirements for New Employees

| Position | Training Requirements |
|---|---|
| Regional Technician | 1. Provide current copies of all network and instrument related information.<br>2. 1 week of intensive training at head office or regional office with emphasis on:<br>• network objectives and scientific rationale for procedures,<br>• instrument service, calibration and related matters,<br>• data recording and submission requirements,<br>• correct sample handling procedures for all aspects of field operations,<br>• sample and data reception and handling procedures,<br>• QC sample submission protocol and rationale.<br>3. Minimum 1 week on-the-job training with another regional technician to learn:<br>• sample collection and handling requirements,<br>• operator training requirements,<br>• in-field instrument servicing,<br>• introduction to all operators and sites,<br>• description and explanation of corrective action procedures,<br>• familiarization with site specific problems or details. |

TABLE 3.2 (cont'd)

Summary of Training Requirements for New Employees

| Position | Training Requirements |
|---|---|
| Regional Technician (cont'd) | 4. Frequent informal telephone contact by network technical coordinator during first 3 months.<br><br>5. Visit by network technical coordinator after 3 months to assess adequacy of training and solve any problems. |

TABLE 3.2 (cont'd)

Summary of Training Requirements for New Employees

| Position | Training Requirements |
|---|---|
| Database Personnel (trained by data-base manager) | 1. Data handling, screening and validation documentation to be provided.<br>2. Existing data handling procedures and programs to be explained.<br>3. All data custody steps to be described including:<br>• data sheet filing,<br>• data entry systems,<br>• data editing,<br>• notification to data manager of problems,<br>• handling QC data submissions and reports,<br>• LIS/SIS systems interaction.<br>4. Data editing procedures to be attempted on dummy or back-up data sets for first several times to avoid accidental data destruction.<br>5. No changes to be made to master data file without making appropriate back-ups.<br>Note: Ideally, new data personnel to be brought on stream in a manner that provides for at least 2 weeks overlap with existing personnel. |

TABLE 3.2 (cont'd)

Summary of Training Requirements for New Employees

| Position | Training Requirements |
|---|---|
| Lab Analyst (trained by laboratory supervisor and other analyst) | 1. Laboratory Operations Manual documenting analytical procedures to be provided.<br>2. Verbal explanation of laboratory objective with respect to APIOS network.<br>3. Explanation of laboratory QA/QC procedures and why they are important - i.e. QC-A and QC-B samples and control charting.<br>4. Step by step explanation of LIS sample preparation and analytical procedures stressing importance of sample contamination.<br>5. Step by step illustration of LIS sample preparation and analytical procedures by experienced analyst.<br>6. Side by side analysis and LIS entry of a "dummy" run with experienced analyst.<br>7. Side by side analysis and LIS entry of a "real" run with experienced analyst.<br>8. Follow up - experienced analyst or laboratory supervisor to scrutinize lab data being generated QC-A and QC-B results check control charts for out of control situations and evaluate new analysts performance.<br>9. Lab supervisor to maintain close, informal contact with new analyst to help solve and avoid problems. |

TABLE 3.3

Summary of Routine Training and Evaluation Requirements by Network Position

| Position | Training Requirements |
|---|---|
| Operator | 1. Assessment and reinforcement training by regional technician every six months as part of internal audits.<br>2. Assessment to be carried out as soon as possible after network changeover.<br>3. Provide operators with periodic feedback about the network (geared to technical level of operators) with network "newsletter" every 6 months to promote communication and maintain operator interest.<br>4. Provide operators with APIOS audio-visual training material as required.<br>5. Regional technician to contact operators frequently on an informal (telephone) basis. |
| Regional Technician | 1. To receive additional or reinforcement training upon request or based on results of annual performance audits to be carried out by network QA Coordinator.<br>2. Technician meetings to be held on a quarterly basis at head office to:<br>- discuss overall network operations,<br>- introduce changes to network,<br>- discuss common field related problems,<br>- provide recommendations to management,<br>- based on field experience, |

TABLE 3.3 (cont'd)

Summary of Routine Training and Evaluation Requirements by Network Position

| Position | Training Requirements |
|---|---|
| Data Management Personnel | 1. Semi-annual assessment and re-training as required. |
| Laboratory Analyst | 1. Assessment should be carried out using internal QC and blind QC samples.<br>2. Monitor laboratory analysts performance using control charts. |

his corrective action duties) for determining what follow up training is required, and for its scheduling.

The performance and adequacy of training for existing personnel is to be carried out in a similar way, but on an annual basis. In this case, external audits may also be useful in identifying areas requiring follow up.

Communication

In a network as large as APIOS it is essential that good communication be established and maintained between all members of the program on both a formal and informal basis. This type of communication is particularly important to maintain the interest of the site operators who, for the most part, have non-technical backgrounds. In order to promote good communication between the operators and the program, two formal communication steps are required.

1) Monthly telephone contact - regional technicians are to telephone operators on a monthly basis in order to ensure all site specific items are in order. The technicians are to prepare a checklist of items to discuss such as:

- instrument status,
- site change,
- inventory,

- sample characteristics (this month),
- problem areas.

This monthly telephone call is to take place just before cumulative samples are to be collected so that it may also serve as a reminder to collect the cumulative samples at the correct time.

2) An APIOS newsletter (oriented to operators) is to be prepared and distributed semi-annually. The objective of the newsletter will be to maintain operator interest in the program and to provide the communication link between the sites and the head office. The newsletter is to contain the following:

- a summary of network stations,
- future network plans,
- recent news in acid deposition research (current events),
- descriptions of on-going special studies,
- quality assurance information and encouragement,
- a summary of network results so that operators can see their contribution to the network.

Informal communication within the network is also essential. Regional technicians are to encourage operators to contact them for any reason, and are to try to coordinate their site visits so that they meet with the operators as often as possible.

## 3.4 Corrective Action

The overall objective of corrective action schemes is to correct or repair areas of malfunction within the network operating system by providing the means to identify and eliminate the causes of malfunction. These corrective action schemes must be well designed, and properly implemented in order to minimize lost or invalid data by providing immediate response to problems or network inefficiencies.

In the short term, the objective of the corrective action scheme is to solve the problem (or notify someone who can) as quickly as possible to avoid lost data, while in the long term the objective is to provide a systematic method for detecting, solving and documenting network problems (recurring, or "one time only" problems) to ensure that they are treated in a timely and systematic manner.

A formal system for reporting and recording corrective action is to be adopted because most problems tend to remain until definite action has been taken by management. The formal corrective action system is to consist of a "closed-loop" system which ensures that the problem is corrected, and verified as corrected. The key steps in the closed loop corrective action scheme are:

i) identify the problem;

ii) assign responsibility for investigating the problem;

iii) determine the cause of the problem;

iv) determine action required to eliminate the problem;

v) assign and accept the responsibility for corrective action;

vi) correct the problem;

vii) verify that corrective action has effectively eliminated the problem;

viii) record all corrective action requirements in a corrective action log.

This formal set of corrective action procedures is of particular importance in view of the regionalization of network field operations since it will provide a sound mechanism for APIOS network management to monitor network operations. In order to follow this scheme, the use of corrective action request forms and a corrective action logging system are to be used. Thus, whenever a problem is noted a corrective action request form is to be completed and distributed as follows:

1) a copy is to be kept by the originator;

2) a copy is to be sent to the immediate supervisor who will be responsible for assigning corrective action duties;

3) a copy is to be sent to the person responsible for implementing the corrective action;

4) a copy is to be sent to the QA Coordinator for insertion into the corrective action log.

A copy of the corrective action form (carbon copy type) is presented in Figure 3.4.

These corrective action forms are to be filled out for all types of problems. It is expected that the majority of the field related corrective action requests will be initiated and carried out by the regional technicians. For example, when a site operator telephones with a problem, the technician is to immediately fill out a corrective action request form. If he is able to correct the problem by "telephone trouble shooting" it should be noted on the form, and then verified by telephone or at the next site visit (as soon as possible). Otherwise an immediate site visit will be necessary to rectify the problem. In both cases, the QA Coordinator is to receive a copy of the corrective action request form as quickly as possible for the documentation in the corrective action log.

FIGURE 3.4

APIOS Corrective Action Form

**APIOS CORRECTIVE ACTION FORM**

FORM NO. _____

Date: _____ Network Operating Area: _____
(lab, data, site etc.)

Circulation: _____

Problem Noted by: _____

Description of Problem: _____

Suggested Corrective Action: _____

Corrective Action Assigned to: _____ by _____

To be completed by (date): _____

Corrective Action Taken: _____

Corrective Action Demonstrated
(Signature) Effective: Yes _____ No _____

Corrective Action Accepted
and Logged by QA Coordinator: _____ Date _____

Follow up Required: _____

Comments: _____

Another extremely important aspect of the corrective action procedures is determining time limits for corrective action to be carried out, and time limits for instrument downtime. The corrective action request form provides an important link since it indicates the date by which repairs must be completed.

These target dates are particularly important as they relate to the field aspects of the program since data lost or invalidated in the field cannot be recovered. Therefore, limits for downtime in each type of monitoring network are to be set at:

1) 5 days for event air and precipitation sampling;

2) 10 days for cumulative air and precipitation sampling.

These limits for downtime must be respected in all cases to meet the strict network objectives for data completeness. In some cases, these limits may require sending out a complete replacement sampling unit to a remote problem site.

## 3.5 Audit Requirements

Quality assurance audits consist of non routine, independent evaluations of the network, its operations and its quality assurance

program. In many ways they may be thought of as "QA on the QA program", or the last chance to spot quality related problems. In general, the objectives of audits are:

1) to evaluate the quality of the network operations, in terms of data accuracy, precision, representativeness, comparability and completeness;

2) to evaluate the effectiveness of the existing quality assurance procedures and systems in meeting the goals of the QA program;

3) to identify areas within the network operations leading to operational or quality related problems.

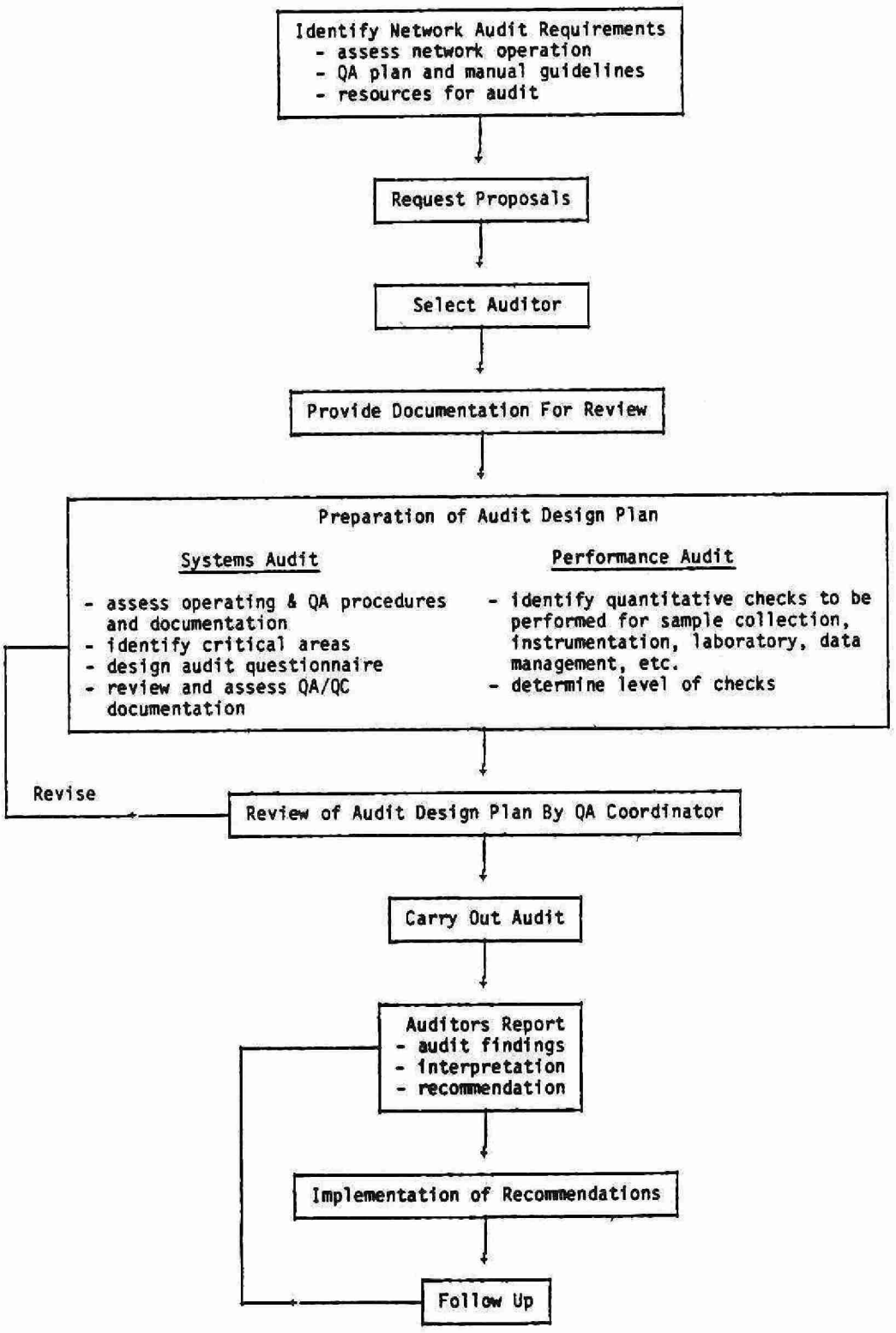
In order to obtain complete and independent assessments of overall data quality, a systems audit (a qualitative assessment of ongoing operational and QA systems) and a performance audit (quantitative comparison of independently collected data on routine network measurements) have been carried out on an regular basis following the general procedure illustrated in Figure 3.5.

In general terms the audits have addressed the following:

- the evaluation of network QA/QC program with respect to objectives stated in the QA plan;

FIGURE 3.5

Recommended Audit Plan Structure



- the site representativeness - evaluate 25% of network sites per year;

- the assessment of documented operational and QA/QC procedures for instrumentation, sample collection and handling, sample analysis and data reduction/reporting for those areas being assessed by the audit;

- an assessment of personnel (operators, regional technicians, data clerks, etc.) relative to documented procedures;

- a summary of action on, or status of recommendations from previous audits.

The specific areas that are to be addressed by the audits are described for each area of network operations in Sections 4, 5 and 6. The key elements of these audits are summarized in Tables 3.4 to 3.7 for network siting, instrumentation, sample collection and handling, and data collection and validation.

Since the majority of sites in the monitoring program have been audited, future external audits will not be carried out on an annual basis. It will be the responsibility of the QA Coordinator and network manager to determine the need for any future external audits.

TABLE 3.4

Site Representativeness Audit Requirements

| Network Area | Operational Element | Checks Required |
|---|---|---|
| Site Representativeness | Site Audit (Visit 25 % of Sites) | - Check accuracy of site documentation, (any omissions, errors or changes).<br>- Check site identification e.g. latitude, longitude, UTM co-ordinates, site/operator address.<br>- Check site details - topography, ground cover, slope, accessibility, windbreaks, land use within 1 km, electrical components.<br>- Check site influences - obstructions, transportation related influences, agricultural activities, local and area pollution sources, population centres.<br>- Evaluate site with respect to siting criteria.<br>- Draw site diagram and take site photographs. |

TABLE 3.5

Instrumentation Audit Requirements

| Network Area | Operational Element | Checks Required |
|---|---|---|
| Instrumentation | Aerochem Metrics | - Configuration checks - instrument security, cleanliness, gasket condition, motor condition, sensor grids, bag fit.<br>- Calibration check - hood movement, measure sensor resistance, bucket orifice, level, orifice height above ground, gasket seal, etc. |
| | Standard Rain Gauge | - Configuration checks - cleanliness of funnel and graduate, security.<br>- Calibration checks - check volume reading with known aliquots, orifice height above ground. |
| | Precipitation Sample Weigh Scales | - Calibration check - with NBS traceable weights. |
| | MIC or Sangamo Type A | - Configuration check - instrument security, cleanliness, gasket condition, motor condition, sensor grids, bag fit.<br>- Calibration checks - hood movement, measure sensor resistance, level, bucket orifice, height above ground, clutch adjustment, heating of sensor, limit switch adjustments, gasket seal, delay time, etc. |
| | Nipher Shielded Snow Gauge | - Calibration checks - proper gauge solutions, orifice height above ground level, orifice diameter, volume measurement with known aliquots, etc. |

TABLE 3.5 (cont'd)

Instrumentation Audit Requirements

| Network Area | Operational Element | Checks Required |
|---|---|---|
| Instrumentation | Metrex Low Vol | - Configuration checks - general instrument condition, cleanliness.<br>- Calibration check - calibrate rotameter, dry gas meter, timer, check filter orifice height above ground, leak test, etc. |
| | Metrex Sequential Sampler | - Configuration checks - general instrument and tower condition, cleanliness.<br>- Calibration checks - calibration of rotameter, dry gas meter, timer, sequenser, leak test, etc. |

TABLE 3.6

Sample Collection and Handling Audit Requirements

| Network Area | Operational Element | Checks Required |
|---|---|---|
| Sample Collection and Handling | Operators | - Performance audit - are most current procedures followed?, contamination possible?, sample transfer performed correctly?<br>- Check sample labelling, storage, inventory, gauge readings, what instrument checks does operator perform?<br>- Are operator data sheets, log books and summary sheets filled out completely and correctly?, etc. |
| | Technician | - Performance audit - are most current procedures followed?, samples processed properly?<br>- field sheets completed properly?<br>- filter loading and unloading, maintenance performed on all instruments regularly, operator performance audit carried out?, QA/QC samples processed routinely? |

TABLE 3.7

Data Collection and Validation Audit Requirements

| Network Area | Operational Element | Checks Required |
|---|---|---|
| Data Collection Validation and Reporting | Operator | - Are operator sheets filled out correctly and completely? Field codes appended properly, all dates, times volumes, etc. recorded? |
| | Technician | - Are field sheets filled out correctly? Monthly summary sheets information transferred correctly from operator sheets? Operator log books examined during site visit? Correct field codes appended properly. Manual data validation checks performed? Check for omission and errors. |
| | Database Manager | - Data input on SIS correctly? Validation checks and computerized data screening carried out properly? Office comments appended properly - systems for validation adequate? Outliers examined? Flags appended? Integrity checks performed? Gross limits applied? |

However, internal audits by the QA Coordinator are to be carried out on regional monitoring operations on a regular basis. Auditing checks such as those in Table 3.7 are to be carried out on an annual basis on regional operations throughout the network. Specific procedures for these internal audits are found in Section 4.2.1.3, "Sample and Data Collection and Handling", and auditing questionnaires are contained in Appendix IV.

Upon completion of the internal audit, a comprehensive report outlining and interpretting the results of the systems and performance audits is to be prepared. In particular, the report is to summarize the results of the evaluations performed and interpret these findings - including strengths and weaknesses of the program - in terms of overall data quality. The report should also include a list of specific recommendations (in order of priority) designed to improve the overall efficiency of the QA/QC or operational procedures employed by the network.

## 3.6 Quality Assurance for Special Studies

Special studies form a large part of the non-routine work carried out by the APIOS network staff. In general, these short-term studies are used to:

1) evaluate and compare new measurement techniques;

2) investigate unusual or interesting trends observed in the network data;

3) investigate specific measurement or sample handling problem areas that may be affecting data quality; and

4) measure specific aspects of data accuracy and precision on a non-routine basis.

Quality assurance is of particular importance in special studies because they are usually of relatively short duration thus increasing the significance of lost or invalid data. Quality assurance plans for special studies are therefore important to ensure that any network modifications resulting from them are based on data that meet the same rigorous quality assurance requirements of routine network data.

Each special, or non-routine survey carried out by APIOS must therefore have a brief quality assurance project plan submitted and approved by the network QA manager prior to the implementation of the study. In this manner, the network will be able to make assessments of the quality of special study data. This special study QA Plan need not be a detailed document, but should include a description of, or reference to, the following QA elements:

- the purpose of the special study to include a description of problem and study objectives;

- the project team and responsibilities;

- sample collection, handling, storage and analysis procedures (references to routine procedures with any deviations noted);

- the project schedule;

- QA procedures to be used for the special study such as,
  - special QA/QC samples to be submitted (number and frequency),
  - special training required,
  - instrument calibration and service procedures and schedules,
  - corrective action procedures (monitoring QA procedures above);

- data reduction and validation procedures;

- data reporting details including,
  - the proposed format,
  - the schedule,
  - QA reporting;

- project costs;
  - in terms of manpower and expenditures.

The time taken to plan and document these quality assurance elements before implementing a special study should facilitate the operation of the study, as well as providing network management with means of assessing the value of the data generated by it.

## 3.7 QA Reporting

The QA Coordinator is to prepare two types of quality assurance reports on a routine basis - reports of data quality to users and quality assurance reports to management.

Quality assurance statistics can be extremely valuable to data users in determining the ultimate uses for the data. Therefore, all future data reports produced by the network are to include a section describing the quality control/quality assurance procedures applied to that particular data set (a synopsis) as well as estimates of the actual quality assurance parameters obtained from the various QA/QC procedures.

The quality assurance information that is to be routinely reported includes:

- a synopsis of the overall QA/QC procedures applied to that particular data set including instrument and operator checks, laboratory QA/QC and data validation and screening;

- statistics on data recovery and reasons for missing or invalid data;

- a summary of analytical procedures employed, their detection limits and how they should be interpretted;

- an indication of the overall level of QA/QC applied to the field and laboratory samples including:
  - duplicate samples,
  - split samples,
  - blank analyses,
  - co-located samplers,
  - prepared samples (field composites and internal lab standards);

- estimates of precision and accuracy for various components of the network based on results of QA/QC checks listed above;

- summaries of results of any on-going network intercomparison studies or laboratory round robins used to assess network comparability;

- summaries of any specific, non-routine QA related items that may influence any particular aspect of the data set (e.g. a site specific contamination problem).

Quality assurance reporting to network management is an essential element of the overall network QA/QC system. QA reports provide the mechanism for the QA Coordinator to report on the status of the network QA/QC system, the effectiveness of past QA/QC actions, and identify QA/QC areas that are to be addressed in the future. By documenting the network QA/QC concerns, the QA Coordinator will be able to obtain management's support in solving outstanding problems and assigning the responsibility to solving problem areas. These reports to management are to be prepared by the QA Coordinator on a annual basis and are to include the following items:

- a summary of data status for all aspects of the networks and statistics on data recovery;
- a summary of the QA/QC activities since the last report (including audits) and an assessment of their effectiveness relative to their cost;
- evaluation of site representativeness based on any site changes during the past year;
- status of outstanding or continuing QA/QC problem areas and a list of recommendations for action (in order of priorities);

- recommendations for changes to QA/QC procedures.

These QA reports to management are an essential component of the overall QA/QC system in that they provide the means of documenting and assessing the effectiveness of the QA program.

## 3.8 Annual Network Review

The operation of the network and its quality assurance systems are to be evaluated by senior network staff on an annual basis. This review is to be held at meetings chaired by the network manager and is to include:

- meetings with air quality chiefs to discuss all field aspects of the monitoring program;
- updates of the network documentation catalogue;
- a review (annual) of the QA report to management;
- an assessment of network site distribution and individual sites (from photographs) for network representativeness;
- a discussion of results of special studies as they relate to sample quality;

- a review of sampling procedures for precipitation and air sampling and analysis.

# 4. FIELD OPERATIONS

## 4.1 Network Representativeness

The representativeness of network siting forms a large part of any network quality assurance program by ensuring that sites are selected and maintained in a manner that meets the overall network objectives, and by providing network data users with the detailed documentation required to make independent assessments of monitoring sites for specific needs. In order to meet and monitor these general objectives of site representativeness, the following specific quality assurance and quality control procedures are to be followed.

1) Rigorous site selection criteria

Site selection criteria for APIOS monitoring networks were originally developed at the implementation stages of the program and have evolved to ensure that the most representative sites possible are used as monitoring locations. The ideal siting criteria to be used for locating new sites, or evaluating existing sites are documented in the APIOS Technical and Operating Manual and include the following requirements:

- there should be no continuous sources of emissions (anthropogenic) within 50 km of the site;

- there should be minimal (preferably none) sources of pollution (air, water, surface transportation), surface storage sources of pollution (sewage plants, landfill sites, fuel depots), or wind activated sources (cultivated fields, sand or salt piles, etc.) within 1 km of the site;

- the sampler should be located on good ground cover (preferably grass covered) and no object should be located within 5 metres of the sampler which extends above the height of the sampler;

- the sampler should be located at a distance of at least 2.5 times the height of any object;

- the site must be accessible to an AC power supply; must also be easily accessible for an operator or technician year-round; and not be susceptible to vandalism;

- there must be an operator on-site or very nearby to collect samples when required; the interest and attitude of this

operator is important in assessing the suitability of the site;

- the site should have surrounding windbreaks whenever possible.

The network has also designed a detailed site evaluation questionnaire to be used in choosing new sites, or carrying out routine evaluations of existing sites. The APIOS Site Description Questionnaire is presented in Appendix I.

2) Site documentation

A detailed set of site descriptions and histories is essential in order to provide data users with the means of assessing the suitability of the data collected at a given site. In particular, this set of documentation is to include the following for all sites;

- a comprehensive description of site characteristics,
- a list of local sampling influences,
- installation and operating details,
- a list of on-site instrumentation,

- a list of advantages and disadvantages of the site (logistical compromises),
- a site history.

This site documentation package is to have a document control system as described in Section 3.2 to ensure that complete and accurate information is maintained at all times and provided to data users upon request.

3) Routine evaluation of sites and networks

Once representative monitoring locations have been selected and established, site and network distribution evaluations are required to assess the ongoing suitability of the sites and their distribution as well as to assess the impact of any site changes on the representativeness of the site. These evaluations are to be carried out at least every two years and are to include the following:

- an assessment of the network site distribution based on routine and non-routine data analysis, network objectives and network resources;

- individual site evaluations based on a current set of photographs illustrating the relationships between instruments and all four compass points;

- site specific data analyses to investigate small scale site and/or instrument representativeness, (these may include, for example, analyses of histograms of sample comment codes for each site to identify recurring problems and analysis of sample characteristics such as $Ca^{2+}$ and $Mg^{2+}$ which may be indicators of a problem with wind blown dust).

These annual network and site evaluations are to be carried out by a team of assessors that includes senior network personnel such as the network manager, data analyst and quality assurance coordinator. When siting or network distribution problems are noted, corrective action is to be initiated, that may include;

- no action, site OK,
- leaving site as is, but flag data,
- upgrade site (upgrading must be specified),
- initiation of a special study to investigate potential problems,

- discontinue or relocate site.

4) Monitor site changes

Whenever any physical, or air pollution source related changes occur in the vicinity of a monitoring site, a "Site Change Form" (Figure 4.1) is to be completed by the regional technician and submitted to the network QA Coordinator. Site operators should be instructed as to the kinds of site changes that are considered important (e.g., physical interferences, such as new buildings or other obstructions, new vehicle access routes, new local pollution sources or industries, changes in nearby land use or other human activity) and they should notify regional technicians who are then responsible for visiting the site as soon as possible to complete a site change form and re-photograph the site. The network QA Coordinator is then responsible for assessing the potential impact of the site changes and updating the site documentation.

This site change form is also to be used to report any other site changes, such as a change in operator or property ownership or other administrative changes.

FIGURE 4.1

APIOS Site Change Monitoring Form

Site: _____

Date of Site Alteration: _____

Date Site Alteration Detected: _____

Describe Nature of Site Change (including physical obstructions changes in human activity or new pollutant sources):

_____

_____

_____

_____

Comments on Potential Impact of Changes:

_____

_____

_____

_____

Form Submitted By _____ Date _____

Site Photo's included? YES____ NO____

Revised Site Plan Sketch on Reverse Side. YES____ NO____

Head Office

Date Site Documentation Updated _____

Corrective Action Required _____

5) External audits

The independent external network audits perform a valuable QA check with respect to network representativeness by 1) assessing the accuracy and completeness of existing network documentation and 2) providing an assessment of site specific and network representativeness based on independent site visits and evaluators. The auditors are to report on any deficiencies in site documentation and provide a value judgement on the representativeness of the site. Once these evaluations have been presented, it is up to network management to decide on appropriate action for sites deemed to be poor or marginal and to provide network resources and commitments.

## 4.2 Network Instrumentation and Sample Handling

This section of the QA manual describes the quality control and quality assurance procedures associated with the instrumentation and field sampling aspects of the various APIOS sampling programs. The general objectives of these sampling programs are to 1) collect representative precipitation and air samples; 2) to maintain the integrity of these samples for subsequent chemical analysis and 3) to collect the ancillary data required for calculating estimates of wet and dry atmospheric deposition.

The objectives of the QA/QC programs associated with these measurement systems are 1) to provide the means of ensuring that the data collected are of sufficient quality to meet the monitoring objectives; and 2) to provide quantitative estimates of data quality in terms of accuracy, precision, completeness, and comparability wherever possible.

The operational aspects of all areas of the field program have been thoroughly documented in the APIOS Technical and Operating Manual which describes the design of each of the measurement systems, the specific instruments employed, the sample collection and handling procedures and the field data reporting requirements. Some aspects of the QA/QC program are also documented in the operations manual.

In this section the field operations aspects of quality assurance are discussed for precipitation and air sampling programs under four general subject areas: systems design, instrumentation QA/QC requirements, sample and data collection quality assurance, and QA/QC monitoring and reporting. The major components that will be discussed in each of these areas are listed below:

1) system design;
   - overview of measurement system;

- summary of "built in" QA/QC;

2) instrumentation QA/QC requirements;
   - manuals and documentation;
   - procurement and pretesting;
   - installation;
   - maintenance and calibration;
   - inventory requirements;

3) sample and data collection quality assurance;
   - quality related sampling precautions;
   - training and evaluation;
   - ancillary measurements;
   - sample submission and preparation;
   - data/sample custody;

4) QA/QC monitoring and reporting;
   - types of checks and samples;
   - schedules;
   - reporting;
   - audits;
   - annual network review requirements.

Due to the similarities in instrumentation and sample handling procedures between the event and cumulative networks, they are discussed together under general headings of precipitation monitoring and air sampling. The differences between event and cumulative sampling are noted where appropriate, and all summary tables are presented separately.

### 4.2.1 Precipitation Monitoring

#### 4.2.1.1 Systems Design

The event precipitation monitoring network employs an Aerochem Metrics type precipitation collector to obtain daily precipitation samples, while the cumulative network employs the MIC Type A wet only collector. These instruments are similar, with the major difference being in the larger diameter collector employed by the event network in order to obtain samples of sufficient volume for chemical analysis of the precipitation collected in one day.

Since bucket type precipitation collectors are generally inefficient (particularly for snow), co-located precipitation gauges are

used to provide measurements of precipitation amount for use in deposition calculations.

The sample collection and handling procedures adopted by the network have been designed with the overall objective of maintaining sample quality and integrity. Many of the sample handling, collection, storage or shipping procedures that have been implemented and documented for the precipitation monitoring networks (sections 4.3.1 and 4.4.1 of the APIOS Technical and Operating Manual) have this underlying quality assurance objective.

4.2.1.2 Instrumental and Equipment QA/QC Requirements

The overall objective of the precipitation monitoring instrumentation is to collect and preserve representative precipitation samples of sufficient volume for complete chemical analysis. In addition, all instruments must be set-up in the same configuration and appropriately positioned at all sites to ensure comparability between sites. It is also essential that all instruments be serviced and calibrated (where possible) on a routine basis to ensure reliability and accuracy. The key areas of quality assurance related to precipitation sampling instrumentation are discussed below.

1) Instrument and equipment procurement and testing

Since the networks have been established for several years, the instruments and procedures for operation are firmly in place. However, some guidelines for instrument procurement, pretesting and initial set-up are useful in the event of network expansions or modifications.

Standard procurement quality control procedures vary widely depending on how complicated and critical the specific instruments or materials are to the operation of the network (USEPA 1976). In general, the instruments and materials used in the APIOS precipitation networks are relatively simple "off-the-shelf" materials rather than special orders or research grade equipment. Therefore, the following procurement procedures are to be followed.

- A current list of suppliers for all network materials including instruments and consumables is to be maintained by the Toronto technician in addition to maintaining detailed specifics of performance characteristics, model numbers, manufacturing details and/or modifications. These specifications are to be explicitly stated in all purchase orders (e.g. dimensions, tolerances, electrical circuitry, etc.).

- Detailed specification of warranty requirements and a commitment by the manufacturer regarding service turn around times, spare parts, delivery dates are to be provided for all equipment purchases.

- Careful inspection upon receipt of equipment and supplies are to be carried out to ensure that specifications are met.

- Acceptance testing and/or "burn in" are to be carried out to to ensure instrument or equipment quality before it is used routinely.

The important aspects of instrument procurement and acceptance testing for the event and cumulative precipitation networks are summarized in Tables 4.1 and 4.2 for the event and cumulative networks, respectively.

Once a new instrument is introduced into the network, an instrument log book is to be started by the Toronto technician to monitor the performance of that instrument over its lifetime.

TABLE 4.1

Event Precipitation Network Procurement and Testing Procedures

| Sampling Component | Procurement Requirements (Purchase Order) | Acceptance Testing Requirements |
|---|---|---|
| Aerochem Metrics Precipitation Collector | - specifications (electrical, bucket, etc.)<br>- warranty<br>- service | - check specified items<br>- laboratory (shop) testing<br>- instrument burn-in-test for 1 week (minimum 3 events) at head office to eliminate "bugs" before sending to field |
| Standard Rain Gauge | - specification (model, stand, etc.) | - calibrate graduated cylinder |
| Nipher Gauge | - specification of dimensions and construction | - measure shield dimensions and calibrate graduated cylinder |
| Sample Bags | - specify construction materials (polyethylene/nylon) and dimensions - with tolerances<br>- specify manufacturer QC<br>- specify packaging requirements to provide convenient quantities for field use with minimum potential for contamination | - measure dimensions of several bags in different lots<br>- submit 9 bags from different lots for chemical analysis<br>- inspect seam quality and bag flaws |

TABLE 4.2

Cumulative Precipitation Network Procurement and Testing Procedures

| Sampling Component | Procurement Requirements (Purchase Order) | Acceptance Testing Requirements |
|---|---|---|
| MIC Type A Precipitation Collector | - specifications<br>- warranty limits<br>- service commitments<br>- desired modifications | - check specified items<br>- shop testing<br>- 1-2 week "burn-in" test period at head office |
| Printed Circuit Boards and Sensors | - specification of important components<br>- circuit diagrams | - testing and adjustment at head office |
| Sample Bags | - specify construction and dimensions with tolerances<br>- specify packaging requirements<br>- specify manufacturer QA | - measure dimensions of several bags from different lots to ensure fit<br>- submit 9 bags for chemical analysis for adsorption or contamination |
| Gauges and Stands | - specify dimensions<br>- provide drawings | - check critical dimensions<br>- check for leaks |

2) Instrument manuals and documentation

Copies of all instrument manuals are contained in the APIOS Instrumentation Binder. The head office copy of this manual is to include all procurement and pretesting information and procedures. This manual also serves as the means for supplementing manufacturers' documentation with specific information required for the APIOS network.

3) Installation and set-up

Once a site has been selected and prepared according to the criteria described in Section 4.1, the actual location of specific instruments must be determined and the instruments installed. The general and logistical items that are to be followed are listed below:

i) instrument should not interfere with other on-site monitoring equipment; this means that all samplers (or meteorological instruments) are to be located so that they are at least 2.5 heights away from other instruments.

ii) ground fault interruptor circuit (GFIC's) are to be installed; receptacle GFIC's are to be used for any locations more than 50 m from the power source or when access to the circuit breaker panel is restricted (e.g. private homes); however, when using a receptacle GFIC, both receptacle outlets are to be protected from moisture or unnecessary power interruptions will result;

iii) windblown contamination must be avoided; this may mean seeding bare soil areas, or arranging for grass trimming in the area of the samplers;

iv) the precipitation monitoring equipment is to be set-up according to specific procedures including:

- Aerochem Metrics sampler orifice are to be 1.5 metres above ground, MIC Type "A" sampler 2.0 metres;

- Standard Rain Gauge are to be 0.5 metres above ground (note grass must be regularly trimmed around the gauge); Nipher gauges are to be 2 metres above ground level;

- all event precipitation sites are to have access to an on-site refrigerator for sample storage.

4) Calibration

The calibration requirements for the precipitation instrumentation are relatively minor since the instruments are quite simple. Calibration procedures are fully documented in the Technical and Operating Manual. The calibration procedures and schedules are summarized in Tables 4.3 and 4.4 for the event and cumulative networks, respectively.

The results of calibrations are to be reported to the QA Coordinator through the use of the instrument log book carbon copy forms that are to be submitted as part of preventive maintenance (next section). These forms are presented in Figures 4.2 and 4.3 for the event and cumulative networks respectively.

5) Preventive Maintenance

Preventive maintenance is defined as an orderly program of actions (e.g. cleaning, adjusting, repairing) designed to prevent the failure of monitoring systems or their parts (USEPA 1976). The overall objectives of preventive maintenance are therefore to 1) maximize data completeness by reducing downtime and to 2) ensure data accuracy by

TABLE 4.3

Event Precipitation Network Calibration Procedures and Schedules

| Measurement Component | Calibration Procedure | Calibration Frequency |
|---|---|---|
| Aerochem Metrics Precipitation Collector | - standardize sensor resistance<br>- check sampler orifice<br>- check level | - every 6 months and after instrument repair or service |
| pH Meters | - standard buffers as described in Technical and Operating Manual | - every day measurements are performed |
| Precipitation Sample Weigh Scales | - standard weights as described in Technical and Operating Manual | - every 6 months |

TABLE 4.4

Cumulative Precipitation Network Calibration Procedures and Schedules

| Measurement Component | Calibration Procedure | Calibration Frequency |
|---|---|---|
| MIC Type A Precipitation Collector | - standardize sensor resistance to 220 KΩ and delay time to 90 ± 30 sec | - every site visit |

FIGURE 4.2

Event Collector Log Book

Year _____

Station # _____ Location _____

Type _____ Serial # _____

| Date | | | | | | |
|---|---|---|---|---|---|---|
| Initials | | | | | | |
| Sensor | | | | | | |
| Hood Gasket | | | | | | |
| Cleanliness | | | | | | |
| Power and Wiring | | | | | | |
| Motor Box | | | | | | |
| Sensor (resistance) | | | | | | |
| Equip. Level | | | | | | |

REPAIRS/COMMENTS

FIGURE 4.3

Cumulative Precipitation Collector Log Book

Year _____

Station # _____ Location _____

Type _____ Serial # _____

| Date | | | | | | |
|---|---|---|---|---|---|---|
| Initials | | | | | | |
| Limit Switches | | | | | | |
| Limit Screw Adjustment | | | | | | |
| Chain Tension | | | | | | |
| Clutch Adjustment | | | | | | |
| Sprockets | | | | | | |
| Expansion Pins, Screws | | | | | | |
| Motor (Serial # _____) | | | | | | |
| Hood Gasket | | | | | | |
| Cleanliness | | | | | | |
| Power and Wiring | | | | | | |
| P.C. Board (time delay) | | | | | | |
| P.C. Board (resistance) | | | | | | |
| Precip. Gauge | | | | | | |
| G.F.I.C. (Test) | | | | | | |

REPAIRS/COMMENTS

maintaining sample integrity. Instrument checks, cleaning and service for precipitation samplers are carried out at four major levels in the APIOS networks. These are:

1) daily/weekly checks and cleaning by operator;
2) quarterly (at minimum) site visits by regional technicians;
3) scheduled or emergency instrument service repair or calibration by regional technicians;
4) repair of major problems by head office personnel.

The details of the instrument maintenance requirements for the operators and technicians are summarized in Tables 4.5 and 4.6 for the event and cumulative networks, respectively.

The corrective action procedures discussed in Section 3.4 above are of particular concern with respect to instrument related problems since they directly affect data capture. If it is not possible to repair or service an existing monitor within the 5 day (event) or 10 day (cumulative) objectives, a complete sampler replacement is to be taken (or sent) to the problem site until repairs can be completed. The use of the corrective action request forms is required as they provide the only formal means of monitoring instrument failure and other field requirements on a continuous basis.

TABLE 4.5

Service and Maintenance Requirements - Event Precipitation Network

| Type of Service | Service Requirements | Service Frequency |
|---|---|---|
| Operator Checks | - check sensor activation and heating with wet finger | - weekly |
| | - check that gasket forms tight seal on bucket | - weekly |
| | - observe hood action during precipitation events | - whenever practical |
| | - check gauges for precipitation | - daily |
| Technician Site Visits (weekly) | - level Aerochem Metrics sampler and all gauges | - monthly |
| | - check hood movement and gasket seal | - each visit |
| | - clean hood gasket and sensor grid | - each visit |
| | - check all electrical connections and Ground Fault Circuit breakers | - monthly |
| | - check obstructions (grass, etc.) around standard gauge | - each visit |
| | - clean standard gauge funnel and cylinder | - monthly |
| | - check Nipher collection container fit | - monthly |
| Scheduled Maintenance | - replace hood gasket | - semi-annually |

TABLE 4.6

Service and Maintenance Requirements - Cumulative Precipitation Network

| Type of Service | Service Requirements | Service Frequency |
|---|---|---|
| Operator Checks | - check sensor activation and heating<br>- check gasket seal<br>- clean sampler hood area, gasket, sensor grids and knife edge collar | - weekly<br>- weekly<br>- every sample changeover |
| Technician Site Visits | - check hood movement, activation and gasket seal<br>- check sensor cleanliness and heating<br>- check hood delay time<br>- check sensor resistance<br>- check inside of instrument for limit switch operation, chain movement, clutch adjustment etc.<br>- check level of instrument and storage gauge<br>- adjust chain tension if required<br>- check ground fault circuitry | - monthly if possible, quarterly at minimum |
| Scheduled Maintenance | - replace limit switches<br>- align chain sprockets<br>- check transformer output and replace if not >12.5 <15 VAC<br>- lubricate hood drive motor<br>- replace hood gasket<br>- clean PC board and sensor grid pins<br>- check and replace internal wiring as required<br>- clean p.c. board edge connectors (eraser) | - every 6 months |

6) Inventory requirements

Another key area of instrument and equipment related QA/QC is the maintenance of field supplies at each site and regional office. It is the responsibility of the head office environmental technician to maintain the overall inventory for both monitoring networks. Regional technicians are responsible for maintaining a 6 month supply of materials to service all their sites, and are to request supplies using the supplies requisition form in Figure 4.4. The head office technician is to send all technicians a notice to fill out these requests on a semi-annual basis (November and May). Site operators are to maintain an inventory of supplies sufficient for at least 2 months. Whenever any supplies are low, the technician is to be notified by using the appropriate section on the operator field sheets or by telephone. Inadequate supplies should never be a reason for lost data.

### 4.2.1.3 Sample and Data Collection and Handling

The general objective of the sample collection and handling procedures for the precipitation samples is to ensure that sample integrity is maintained throughout all stages of sample collection, handling and shipping procedures. In order to meet this general monitoring objective, there are a number of more specific quality assurance objectives that are described in detail below.

FIGURE 4.4

Supplies Requisition Form

Region: _____
Name: _____
Date: _____

Six Month Supplies Request

| Item | # Required |
|---|---|
| Cable Ties / Twist Ties | |
| Ethanol | |
| Kimwipes | |
| Gloves | |
| Tape | |
| Labels | |
| Whirl-Pak Bags: 18 oz | |
| 16 oz | |
| Zip-Lock Bags | |
| P.C. Boards Aerochem/MIC | / |
| Sensor Grids Aerochem/MIC | / |
| Limit Switches | |
| Thermometers | |
| Gaskets Aerochem/MIC | / |
| Tweezers | |
| Squeeze Bottles | |
| Event Bottles | |
| Bags: Aerochem | |
| MIC Short | |
| MIC Long | |
| Filters: Nylon | |
| Teflon | |
| W40 | |
| W41 | |
| Field Sheets: Precipitation | |
| Air | |
| Operator Sheets: Cumulative | |
| Event | |
| Stationary | |
| Pens | |
| Log Books (specify) | |
| Other | |

1) The first objective is ensuring that sample collection, handling and shipping procedures are well designed and properly implemented.

In order to achieve this primary objective, the network has used its experience in past precipitation monitoring networks to develop a detailed set of operating procedures designed to maintain sample quality and operational efficiency. These procedures have been documented along with illustrative diagrams in the Technical and Operating Manual. Some of the key steps to ensure sample integrity, that are stressed throughout Technical and Operating Manual include:

- designing operator handling procedures to minimize potential sample contamination by:
  - not leaning over exposed collector while changing bags;
  - standing downwind of open collectors when possible;
  - keeping hands and arms out of collectors while changing bags;
  - wearing <u>new</u> gloves whenever the inside bag surface is touched;
  - following explicit instructions for transfer of event samples to bottles to avoid contamination from the

exterior of the bag and to ensure all the material is transferred;

- ensuring chemical stability of event samples by storing them at ~4°C in on-site refrigerators (note this is not used for cumulative samples since they sit at ambient temperatures for several days in the collectors) and shipping them in coolers with ice packs.

- protecting samples during shipment by proper packing, and sample inspection before shipment (e.g. event samples are transferred to bottles, while cumulative samples are sealed using on-site heat sealers);

- measurement of field pH is done as quickly as possible for event samples to provide a pH measurement before any significant sample changes occurs.

The networks are to continue to refine and update the network operating procedures through the use of special studies (to document potential problem areas such as sample storage, sample handling, sorption effects of bags, dry deposition during precipitation, etc.)

and analyses of past operating problems (e.g. summary statistics on reasons for lost data, etc.).

2) The second major objective involves ensuring that the documented operating procedures are properly executed

The most important aspect of this quality assurance objective is ensuring that all operators and technical staff have been properly trained and evaluated as described in Section 3.3. In particular, operators are to be evaluated by the regional technicians informally on a semi-annual basis in addition to ongoing checks that occur at each visit or whenever data and samples are submitted. When problems are observed, the operators are to be retrained by the technician, and re-evaluated within 3 months. It is also essential that all members of the program receive proper documentation through the documentation control system described in Section 3.1 above.

These areas of sample collection and handling are to be further monitored through on-site internal performance audits by the QA Coordinator. Auditing questionnaires for these internal audits are contained in Appendix IV. Each region is to have a selected number of sites audited in this fashion on a annual basis. A findings report is to be prepared and submitted to the network manager and air quality

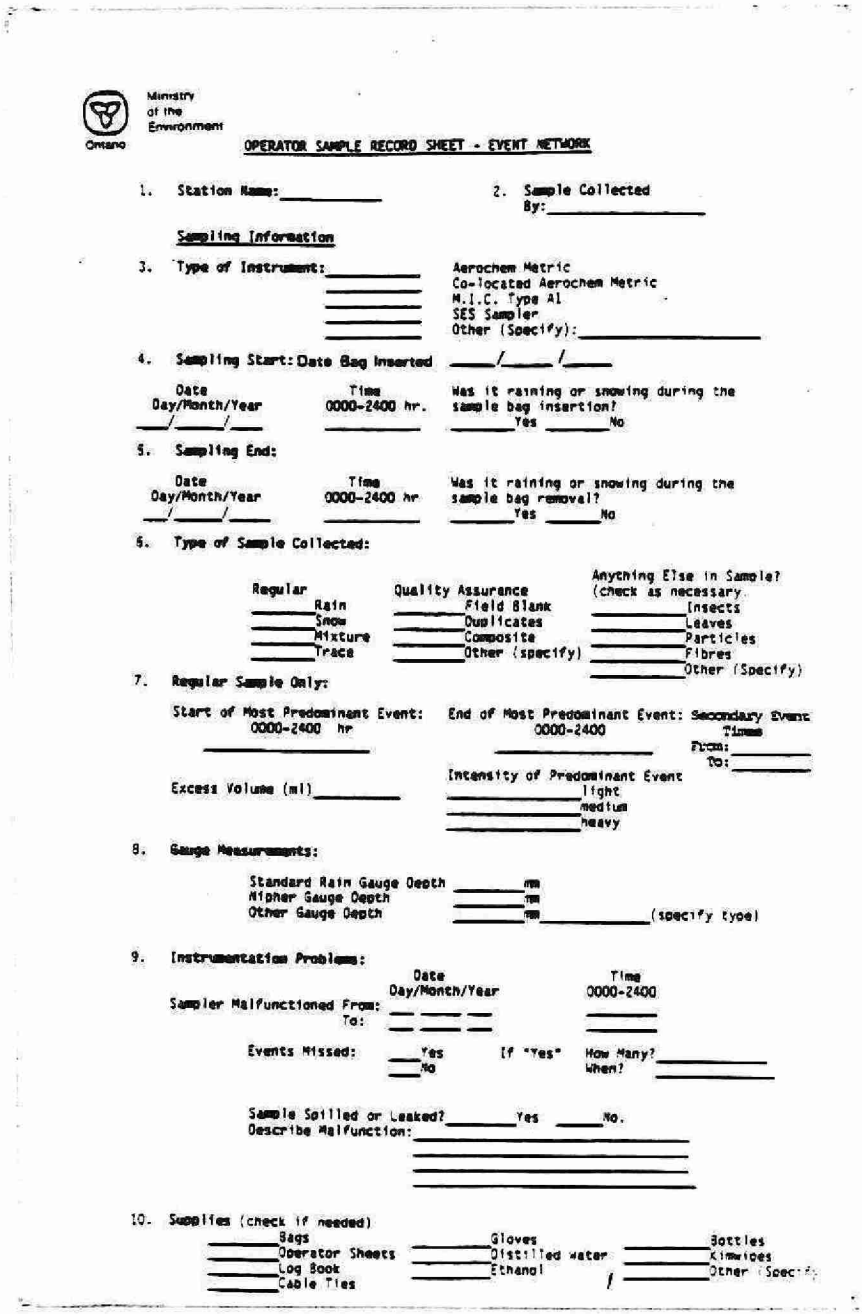
chief of the audited region. Corrective actions as a result of this evaluation are to be acted upon by the regional technician.

3) The third major objective requires all relevant sampling information to be properly recorded.

This is an important QA objective. Care has been taken to ensure that operator and technician sample submission sheets (Figures 4.5, 4.6 and 4.7) are designed to permit simple recording of all sample information. Regional technicians are responsible for transferring information from the operator sheet to the field submission form that accompanies the samples to the laboratory. It is the technician's responsibility to carefully transcribe and evaluate the information on these forms, and to make sure that all relevant information is passed on. The technician is to telephone operators immediately in the event of missing or suspicious information. It is at this stage that technicians perform the first quality control check by completing the technician field summary data sheets and carrying out a manual "common sense" screening of the data. In the case of the event network, an Event Network Summary Sheet (Figure 3.1) is completed on a monthly basis and submitted to the QA Coordinator. This sheet provides a simple means for the technician to summarize all event samples in his region and

FIGURE 4.5

## Operator Sample Record Sheet - Event Network



Ministry of the Environment
Ontario

OPERATOR SAMPLE RECORD SHEET - EVENT NETWORK

1. Station Name:_____ 2. Sample Collected By:_____

Sampling Information

3. Type of Instrument:_____
   - Aerochem Metric
   - Co-located Aerochem Metric
   - M.I.C. Type A1
   - SES Sampler
   - Other (Specify):_____

4. Sampling Start: Date Bag Inserted ____/____/____

| Date Day/Month/Year | Time 0000-2400 hr. | Was it raining or snowing during the sample bag insertion? |
|---|---|---|
| ___/___/___ | _____ | _____Yes _____No |

5. Sampling End:

| Date Day/Month/Year | Time 0000-2400 hr | Was it raining or snowing during the sample bag removal? |
|---|---|---|
| ___/___/___ | _____ | _____Yes _____No |

6. Type of Sample Collected:

| Regular | Quality Assurance | Anything Else in Sample? (check as necessary) |
|---|---|---|
| _____Rain | _____Field Blank | _____Insects |
| _____Snow | _____Duplicates | _____Leaves |
| _____Mixture | _____Composite | _____Particles |
| _____Trace | _____Other (specify) | _____Fibres |
| | | _____Other (Specify) |

7. Regular Sample Only:

Start of Most Predominant Event: 0000-2400 hr _____

End of Most Predominant Event: 0000-2400 _____

Secondary Event Times From:_____ To:_____

Excess Volume (ml)_____

Intensity of Predominant Event
- _____light
- _____medium
- _____heavy

8. Gauge Measurements:

- Standard Rain Gauge Depth _____mm
- Nipher Gauge Depth _____mm
- Other Gauge Depth _____mm_____(specify type)

9. Instrumentation Problems:

| | Date Day/Month/Year | Time 0000-2400 |
|---|---|---|
| Sampler Malfunctioned From: | ___ ___ ___ | _____ |
| To: | ___ ___ ___ | _____ |

Events Missed: ___Yes ___No If "Yes" How Many?_____ When?_____

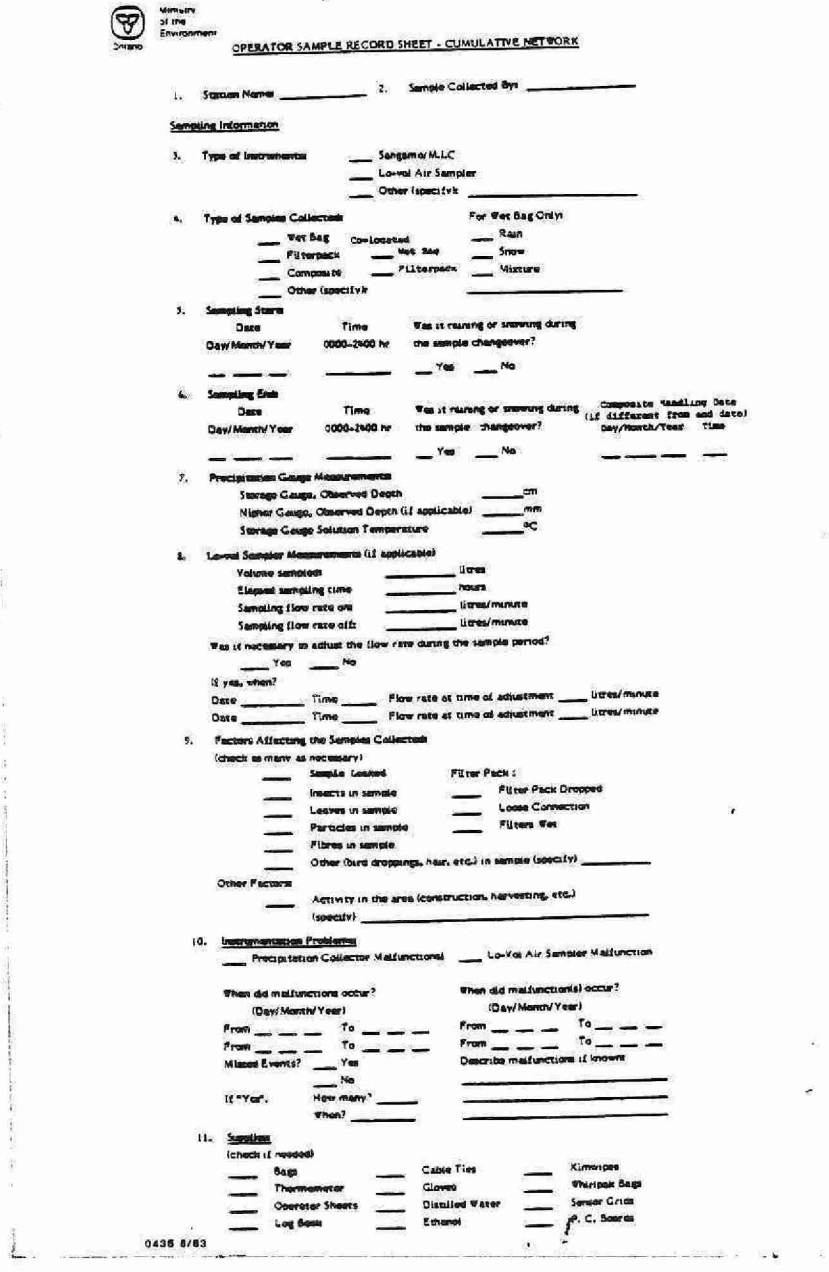
Sample Spilled or Leaked?_____Yes _____No.
Describe Malfunction:_____

10. Supplies (check if needed)

| | | |
|---|---|---|
| _____Bags | _____Gloves | _____Bottles |
| _____Operator Sheets | _____Distilled water | _____Kimwipes |
| _____Log Book | _____Ethanol | _____Other (Specify) |
| _____Cable Ties | | |

FIGURE 4.6

## Operator Sample Record Sheet - Cumulative Network



Ministry of the Environment
Ontario

OPERATOR SAMPLE RECORD SHEET - CUMULATIVE NETWORK

1. Station Name _____ 2. Sample Collected By: _____

Sampling Information

3. Type of Instruments
___ Sangamo M.L.C
___ Low-vol Air Sampler
___ Other (specify): _____

4. Type of Samples Collected
___ Wet Bag
___ Filterpack
___ Composite
___ Other (specify): _____
Co-Located
___ Wet Bag
___ Filterpack
For Wet Bag Only:
___ Rain
___ Snow
___ Mixture

5. Sampling Starts
Date: Day/Month/Year ___ ___ ___
Time: 0000-2400 hr _____
Was it raining or snowing during the sample changeover? ___ Yes ___ No

6. Sampling Ends
Date: Day/Month/Year ___ ___ ___
Time: 0000-2400 hr _____
Was it raining or snowing during the sample changeover? ___ Yes ___ No
Composite Handling Date (if different from end date): Day/Month/Year ___ ___ ___ Time ___ ___

7. Precipitation Gauge Measurements
Storage Gauge, Observed Depth _____ cm
Higher Gauge, Observed Depth (if applicable) _____ mm
Storage Gauge Solution Temperature _____ °C

8. Low-vol Sampler Measurements (if applicable)
Volume sampled _____ litres
Elapsed sampling time _____ hours
Sampling flow rate on _____ litres/minute
Sampling flow rate off: _____ litres/minute
Was it necessary to adjust the flow rate during the sample period?
___ Yes ___ No
If yes, when?
Date _____ Time _____ Flow rate at time of adjustment _____ litres/minute
Date _____ Time _____ Flow rate at time of adjustment _____ litres/minute

9. Factors Affecting the Samples Collected
(check as many as necessary)
___ Sample Leaked
___ Insects in sample
___ Leaves in sample
___ Particles in sample
___ Fibres in sample
___ Other (bird droppings, hair, etc.) in sample (specify) _____
Filter Pack:
___ Filter Pack Dropped
___ Loose Connection
___ Filters Wet
Other Factors:
___ Activity in the area (construction, harvesting, etc.) (specify) _____

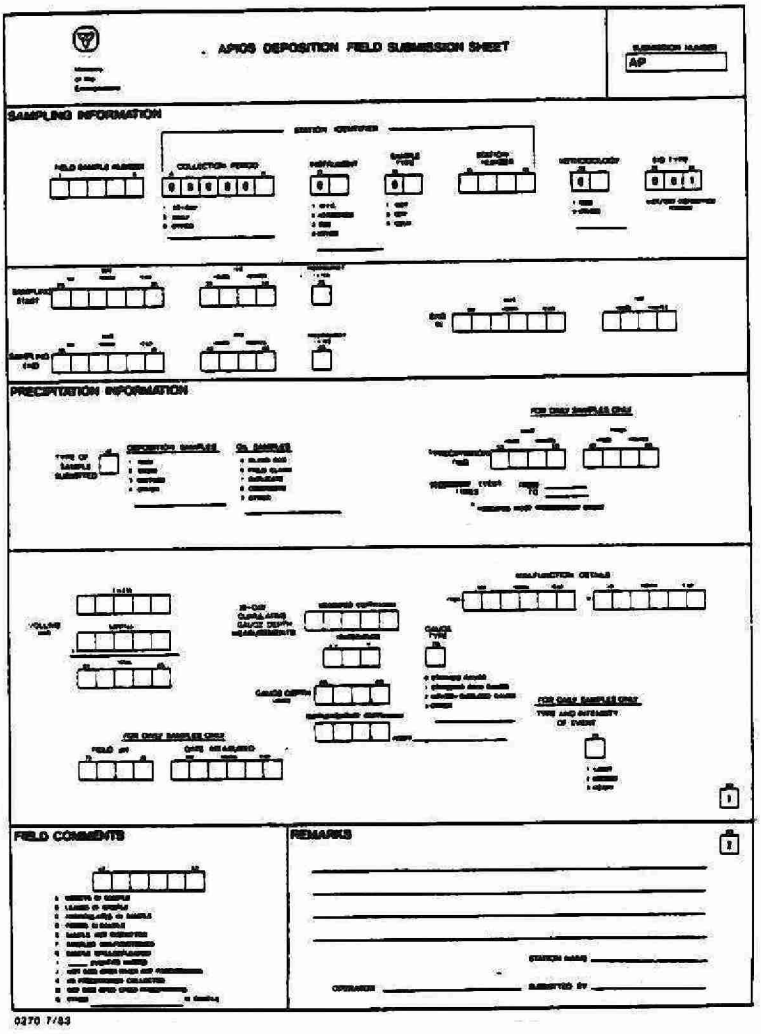
10. Instrumentation Problems
___ Precipitation Collector Malfunctioned ___ Lo-Vol Air Sampler Malfunction
When did malfunctions occur? (Day/Month/Year)
From ___ ___ ___ To ___ ___ ___
From ___ ___ ___ To ___ ___ ___
Missed Events? ___ Yes ___ No
If "Yes", How many? _____ When? _____
When did malfunction(s) occur? (Day/Month/Year)
From ___ ___ ___ To ___ ___ ___
From ___ ___ ___ To ___ ___ ___
Describe malfunctions if known: _____

11. Supplies
(check if needed)
___ Bags
___ Thermometer
___ Operator Sheets
___ Log Book
___ Cable Ties
___ Gloves
___ Distilled Water
___ Ethanol
___ Kimwipes
___ Whirlpak Bags
___ Sensor Grids
___ P. C. Sheets

0436 8/83

FIGURE 4.7

APIOS Deposition Field Submission Sheet



check that all stations are reporting. Some of the simple checks that are to be performed by the technician include:

- check that all stations usually report events on the same day (spatial variability is to be expected);
- provide a summary of QA/QC samples submitted (discussed later);

A similar form (Figure 3.2) is to be used in the cumulative network, and is to be submitted to the QA Coordinator on a monthly basis. Technicians are to carefully examine the cumulative samples and their data sheets to ensure all information relating to sample quality is recorded. The importance of sample comment codes etc., must be stressed to operators and checked by telephone when not properly documented. Operators are to be encouraged to report as much information as possible on the data sheets.

4) The fourth main objective is to ensure that all instrumentation works properly and all appropriate information is recorded.

To meet this objective, it is essential that all information reported by the technicians (submission sheets, summaries, etc.) be

examined and evaluated on a continuous basis. Therefore, submission sheets are to be checked by the database clerk for proper codings and completeness. The QA Coordinator is also to examine and evaluate all summary sheet information (event and cumulative) and specify corrective action as required through the use of corrective action forms (Fig. 3.4).

#### 4.2.1.4 QA/QC Parameters for Precipitation Collection Programs

There are a number of specific quality control and quality assurance procedures that are to be carried out on the precipitation collection network of the APIOS program. These procedures are discussed below, and summarized in Tables 4.7 and 4.8 for the event and cumulative networks, respectively.

1) Data precision

Precision is defined as a measure of the mutual agreement among individual measurements of the same property (USEPA, 1976). For precipitation monitoring, imprecision may occur as a result of sample variability introduced from:

TABLE 4.7

Summary of Routine QA/QC Sampling and Procedures for the Event Precipitation Network

| QA/QC Sample or Procedure Type | Quality Assurance Parameter Measured | Frequency |
|---|---|---|
| Co-located Sampling (Aerochem Metrics and Standard Gauges) | - overall site/sample precision (instrument, operator, shipping and lab precision) | - all events, 1 site per region |
| Duplicate/Split Field Samples | - analytical variability | - 1 large volume event per 1 month per site |
| Bag Blanks | - sample accuracy relative to sample contamination | - 1 bag per quarter per region |
| Bag QA | - procurement QA | - 9 bags from each new batch |
| Dry Bag Blanks (bag that was in collector for minimum of 2 dry days) | - sample accuracy relative to sample contamination | - 1 bag/site/month |
| Composite Precipitation Samples (400 ml) | - sample accuracy relative to volume determination, container effects and potential contamination | - 1/quarter at each site |
| Event Sample Volume | - accuracy of volume determination by regional weigh scales | - 1/quarter in each region |
| Field pH Measurement | - accuracy of field pH meters<br>- analysis of dilute $HNO_3$ solution | - 1/quarter in each region<br>- 1/quarter in each region |

TABLE 4.8

Summary of Routine QA/QC Sampling and Procedures for the Cumulative Precipitation Network

| QA/QC Sample or Procedure Type | Quality Assurance Parameter Measured | Frequency |
|---|---|---|
| Co-located Samples (MIC and Precipitation Gauge) | - overall site/sample precision | - continuous 1 site per region |
| Bag Blanks | - sample accuracy relative to sample contamination | - 1 bag per quarter per region |
| Bag QA | - procurement QA | - 9 bags from each new batch |
| Composite Precipitation Samples (1,000 mL) | - sample accuracy relative to sample volume, container effects and contamination | - twice per year at each site |

i) equipment variability including instrument sensing characteristics and sample container variations;

ii) operator variability resulting from operator handling;

iii) variability introduced in sample storage and/or shipment;

iv) analytical variability.

To obtain a measure of the overall variability (i to iv above) each of the precipitation monitoring networks operates a duplicate set of collectors and gauges at one site in each region to collect "co-located" samples of the same precipitation event(s). In this way, the results obtained by the co-located samplers can be compared to provide estimates of the overall precision of precipitation sampling within each region.

Imprecision introduced by sample handling and analysis procedures is to be estimated by sample splitting as described in the Technical and Operating Manual for event samples. Once per month at each event site, one large volume event is split by the site operator into three samples. In this way, the instrument and operator variabilities

are eliminated (assuming sample homogeneity) and an estimate of the handling and analytical precision may be obtained.

Other estimates of analytical precision carried out by laboratory personnel are obtained through the use of split samples, replicated analyses etc. as described in Section 5 of this manual.

2) Data accuracy

Accuracy is defined as the degree of agreement of a measured value with an accepted reference or true value (USEPA, 1976). At the present time, it is not possible to measure the absolute accuracy of wet deposition data; however, it is possible to measure certain aspects of data accuracy.

The laboratory operations determine the analytical accuracy through the use of calibration standards, quality control samples and other prepared samples as described in Section 5.3.2.

Field accuracy is monitored through the used of field blank sample analysis and the analysis of prepared samples subjected to operator handling to identify potential areas of sample deterioration or contamination. Two types of blank samples are used in both the

event and cumulative network, and the detailed sampling and submission procedures are described in the Technical and Operating Manual.

Bag blanks consist of unused sample bags submitted quarterly for blank analysis using deionized water at the laboratory. These checks provide on-going quality assurance on bag quality and serve to identify contamination problems. In addition, several (nine) new bags are submitted for chemical testing from each new batch obtained from the manufacturer. These types of bag analyses are to be carried out, reported and interpreted by the QA Coordinator as quickly as possible to ensure that faulty or contaminated bags are not used in the field collectors.

Since event sample bags are not changed on days in which no precipitation occurs, the event network has established a test in which a dry bag is submitted (and extracted with deionized water) to examine the contribution of dry deposition or contamination to the precipitation samples. Operators are to submit one bag per month that has sat in the collector for a minimum of two days without precipitation. Detailed procedures for the collection and submission of these field blanks are described in the Technical and Operating Manual. Regional technicians are responsible to ensure that all operators submit these samples on a regular (monthly) basis.

Another type of sample accuracy check that is performed in both networks consists of the use of prepared "composite" precipitation samples to assess contamination, sorption and sample deterioration effects throughout the measurement system (these include sampler bag and container effects, operator handling and shipping to the laboratory). The composite precipitation samples are generated by the laboratory and distributed to operators in polystyrene bottles in specific quantities. The samples are poured into sample bags and then treated as routine samples. In this manner, samples of known composition and volume are processed by the system and any deterioration or contamination is noted. Procedures for composite sample submission are contained in the Technical and Operating Manual.

3) In-field measurements

In each of the precipitation monitoring networks, there are ancillary measurements taken in the field associated with the precipitation samples. The relevant routine QA/QC checks are described below.

i) The sample volume measurement is evaluated on a regional basis every quarter by the head office technician sending premeasured water samples to each region for volume determination by measurement of the weight of the sample. The results are to be reported on the APIOS Weighing Scale Audit Sheet (Figure 4.8) and sent immediately to the QA Coordinator. Corrective

action, if required, is to be initiated by the QA Coordinator upon receipt of the weighing results.

ii) The accuracy of regional pH meter determination of field pH is checked on a quarterly basis in each region using laboratory prepared samples (pH 3.5-4.5) prepared with dilute $HNO_3$. The results are reported on the APIOS Field pH Measurement Check report form (Figure 4.9). The head office technician is responsible for arranging with laboratory personnel to prepare and send the solution to the regional offices (excluding NW and NE). Upon completion of the pH measurment, the regional technician is to send the completed report form to the QA Coordinator who will then be responsible for initiation and correction actions, if required.

3) Data comparability

Data comparability is the measure of the confidence with which one data set can be compared with another. The network procedures that have been designed to meet this objective include:

- the use of standard units in all reports;
- the routine calculation and reporting of accepted QA parameters such as collector efficiency, anion/cation ratio, etc.;

FIGURE 4.8

APIOS Weighing Scale Audit Sheet

| APIOS Weighing Scale Audit Sheet<br>**Audit Dates** _____<br>**Serial Number:** _____<br>**Location:** _____<br>**Name:** _____ | | |
|---|---|---|
| # (Weigh 3 Times) | Digital Readout (grams) | Comments |
| 1. | | |
| 2. | | |
| 3. | | |
| | Average: | |
| | Stand. Dev: | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| Office Use<br>**Action Required** | | |
| **Action Taken:** | | |

FIGURE 4.9

APIOS Field pH Measurement Check

APIOS Field pH Measurement Check

Date: _____

Regional Office: _____

$HNO_3$ I.D.#: _____

pH Measurements Date of Measurements: _____

1. _____

2. _____

3. _____

Average: _____

Stand. Dev.: _____

Action Required: _____

_____

_____

_____

_____

Action Taken: _____

_____

_____

_____

_____

- the use and documentation of routine data flags, comment codes, etc.;

- participation in inter-laboratory round robin programs;

- co-location of monitors with equipment from other similar networks.

It is important that APIOS personnel keep informed about the programs of other networks in order to maintain this high degree of data comparability. It is also essential that data collected from sites co-located with other networks (e.g. Dorset, Fernberg) be reported and evaluated on a regular and routine basis.

4) Data representativeness

The network representativeness assessment procedures described in Section 4.1 are essential. The use of annual network site and data evaluations by senior network personnel cannot be overemphasized.

5) Data completeness

Data completeness objectives have been set requiring chemical data from 90% of all samples. Regional technicians are to monitor sample collections at each site on a routine basis in order to achieve the data completeness objective. The 5 day/10 day downtime criteria for event and cumulative networks respectively are not to be exceeded.

6) Monitoring QA/QC parameters and requirements

The large number of QA/QC tasks being implemented into the APIOS monitoring activities will significantly increase the demands placed on the regional technicians and the QA Coordinator. A QA/QC activity monitoring system is to be set-up for each region by the QA Coordinator. Presented in Figure 4.10 is a QA/QC monitoring log. By monitoring the number, frequency, and type of QA/QC field samples on a regional basis, the QA Coordinator can monitor the effectiveness and completeness of the QA/QC system in each region.

Corrective action is to be taken by the QA Coordinator whenever samples have not been collected or procedures have not been followed.

7) QA/QC Data Reporting

Routine reporting and interpretation of QA/QC results is essential. It is important that estimates of accuracy, precision, comparability and completeness be reported in a manner that facilitates their use by network management in determining the effectiveness of the monitoring and QA/QC programs. Methods for manipulating and reporting these data are described in Table 4.9. These data calculations are to be included in all data quality reports.

FIGURE 4.10

APIOS Site Status QA/QC Monitoring Log

Region: _____

Sample Type: _____

| Site | Jan | Feb | Mar | Apr | May | Jun | Jul | Aug | Sep | Oct | Nov | Dec |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |
| Status | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- | ------- |
| QA/QC | | | | | | | | | | | | |

Sampler Status
xxxxxxx sampler up all month
——— sampler down part of month
------- sampler down all month

QA/QC Time Table
0 composite received
* blank bag received
+ calibration carried out
± service carried out
• operator site audit required
etc.

□ field blank received

TABLE 4.9

QA/QC Data Reporting Requirements for Precipitation Sampler

| QA/QC Procedure Type | QA Parameter of Interest | Recommended Reporting Method |
|---|---|---|
| Co-located Sampler | Overall precision | 1) Range of % difference by events<br>2) Mean % difference<br>3) Interpretation of significance of 1 and 2 |
| Duplicate/Split | Laboratory precision | 1) Range of % differences<br>2) Mean % differences<br>3) Interpretation |
| Bag QA | Procurement QA | 1) Levels of all parameters<br>2) Significance of results |
| Bag Blanks | Potential contamination | 1) Site by site results for all parameters<br>2) Interpretation of significance |
| Composite Precipitation Samples | Sample accuracy relative to container effects and contamination | 1) Site by site results for all parameters<br>2) Interpretation |
| Field pH Measurement (event only) | Accuracy of field pH | 1) % difference by region<br>2) Interpretation |
| Sample Volume Evaluation | Volume accuracy | 1) % difference by region |
| Field Blanks (Dry bag) | Sample contamination | 1) Raw data results<br>2) Interpretation |
| Internetwork Co-location | Comparability | 1) Results<br>2) Interpretation |

4.2.2 Air Sampling

4.2.2.1 Systems Design

The daily dry deposition network utilizes a Metrex SAS8-25 sequential air sampler to collect gaseous compounds and particulate matter through a multi-stage 47 mm filter pack at 10 m above the ground sampling air at approximately 25 lpm. The sequential sampler switches automatically through 8 ports with each port open for a 24 hour period. The sample housing also holds a blank filter for passive correction. The air volume sampled is monitored by a temperature compensating dry gas meter coupled to 8 total flow counters. Individual filter packs are loaded with 3 types of filters, each type specific for certain pollutants. The first filter is a 2 µm Teflon filter which collects particulate sulfate, nitrate and ammonium. The Teflon filter is followed by a 1 µm nylon filter, used for the selective adsorption of vapour phase nitric acid. The final stage contains two Whatman 41 cellulose filters impregnated with a potassium carbonate/glycerol solution for sampling of sulfur dioxide. This system provides daily integrative measurement of these gaseous and particulate compounds.

In contrast, the cumulative dry deposition network utilizes a Metrex AS-2 low volume air sampler. The two stage 47 mm filter packs are mounted 2 m above the ground and sample for 28 days at a rate of approximately 2 lpm. The volume is determined by a temperature compen-

sated dry gas meter coupled to a digital volume counter and a timer. The individual filter packs are loaded similarly to those used in the daily dry network except a Whatman 40 filter is used in place of a Teflon filter to collect particulate matter for analysis of ions and metals. This change is required because of the higher loading over a 28 day period which brings about an undesirable pressure drop across the Teflon filter.

The only differences between event and cumulative samples are the sampling period (1 vs. 28 days), the sample volume and the particulate sampling medium. The sequential samplers are located at event sites and are generally operated by regional technicians (except Fernberg) while the low volume samplers are located at cumulative sites and are operated by site personnel.

Since the two types of sampling are almost identical, they will be discussed together with important differences identified.

The sample collection and handling systems for the event and cumulative dry samples have been designed to maintain sample quality and integrity, and are described in detail in the Technical and Operating Manual. Some of the key QA/QC steps built into the system include:

- all Whatman filters are prepared (cut, and/or impregnated, etc.) in a "clean room" at head office every 3 months;

- all filter packs are cleaned, loaded and packaged under carefully controlled conditions in the regional offices to limit potential sample contamination;
- detailed procedures are documented to ensure contamination is avoided during filter preparation, cutting and loading;
- filter storage is tightly controlled and monitored;
- unambiguous labelling systems are used to avoid labelling errors;
- all filter packs are separately packaged during transport to avoid cross contamination;
- passive filters and handling blanks (described below) are used to monitor sample contamination;
- detailed data recording procedures are documented to ensure all appropriate data are reported;
- routine instrument calibrations are carried out to check flow meter accuracy, and procedures are in place to incorporate calibration results;
- routine service and maintenance are carried out to minimize lost data due to breakdown; and
- appropriate QA/QC sampling has been incorporated to monitor data quality.

The above points represent a summary of some of the key QA/QC steps built into the APIOS air sampling programs. The specifics of the instrument, related sample collection and handling and QA/QC monitoring are described in the following sections.

4.2.2.2 Air Sampling Instrumentation QA/QC

The air sampling instrumentation monitoring and quality assurance objectives are basically the same as those for precipitation sampling; to collect and preserve representative air sampling filters for subsequent chemical analyses. Instruments must therefore, be set up and operated to meet the network requirements for collecting precise, accurate, comparable and representative samples. Many of the general discussions describing quality assurance requirements for precipitation instrumentation in Section 4.2.1.2 also apply for the air sampling equipment. The important areas of air sampling instrument related QA/QC are summarized and specific requirements are tabulated below.

1) The main objective of the instrument equipment and supplies procurement and pretesting is to ensure that the stated quality and specifications of these items are met before they are incorporated into the network operating system, and to test each new instrument for an appropriate period of time before it is put into routine operation. The important requirements for instrument procurement and pretesting for air sampling equipment are summarized in Table 4.10.

As in the precipitation monitoring systems, all instrument (samplers and associated calibration equipment) documentation and

TABLE 4.10

Air Sampling Instrument and Equipment Procurement and Pretesting Requirements

| Sampling Component | Procurement Requirement (Purchase Order) | Acceptance/Pretesting |
|---|---|---|
| Metrex #AS2 Low Volume Sampler | - specifications<br>- warranty limit<br>- service commitment<br>- desired modification | - burn in test at head office 1 week, eliminate bugs, calibrate |
| Metrex #SAS8 Sequential Sampler | - specifications<br>- warranty limits<br>- service commitment<br>- desired modification | - burn in test at head office 1 week, eliminate bugs, calibrate timer flow counter |
| Swinnex 47 mm Filter Packs | - specification<br>- drawing | |
| Filters | - specification of dimensions, pore size, type | - submit 5 from each lot to laboratory for chemical analysis |
| Associated Hardware<br>- stands<br>- filter holders<br>- tubing | - specify dimensions<br>- construction material<br>- tolerance levels<br>- tubing type, size, thickness | - N/A |
| Calibration Equip.<br>- Hasting 0-10 lpm<br>- Hasting 0-50 lpm | - certificate of calibration to NBS standards | - Check calibrations, drift stability, etc. |

manuals are to be kept up to date in the technical instrument binders. This documentation is to include instructions for calibrating the flow monitoring equipment used to calibrated the field monitors.

2) Calibration

The calibration requirements for the air sampling instrumentation are more stringent than those for the precipitation monitoring systems. Since the accuracy of air concentration measurements is directly determined by the volume reported by the instrument, it is essential that the flow meters and counters be monitored on a regular basis to ensure valid correction factors are being applied to the data. The accuracy of these calibrations can only be ascertained by using primary standards (e.g. mass flow meters) that are traceable to acceptable reference standard procedures such as soap bubble flow meters. The calibration procedure for the monitors and mass flow meters are described in Section 4.6 of the Technical and Operating Manual and are summarized in Table 4.11.

The results of these air sampling calibrations are to be immediately reported to the QA Coordinator through the use of a calibration sheet as illustrated in Figure 4.11. In addition, the regional technician is to maintain a record book of instrument calibration

TABLE 4.11

Air Sampling Calibrations and Schedules

| Instrument | Calibration Procedure | Frequency |
|---|---|---|
| Metrex # AS2 Low Volume Sampler | Calibrate rotameter and temperature compensated dry gas meter with mass flow meter as described in operations manual. | Every year and after instrument repair or service. |
| Metrex # SAS8 Sequential Sampler | Calibrate rotameter and temperature compensated dry gas meter as described in operations manual. | Every 6 months and after instrument repair or service. |
| Hasting 0-10 lpm flow meter | Check calibration using a primary standard (bubble meter) | Every year or after instrument repair or service. |
| Hasting 0-50 lpm flow meter | Check calibration using a primary standard. | Every year or after instrument repair or service. |

FIGURE 4.11

**APIOS Air Sampler Calibration Sheet**

**Calibration Date:** _____

**Date of Last Calibration:** _____ **% Error:** ± _____ (last calib.)

**Instrument Type:** _____

**Serial Number:** _____

**Station Number and Location:** _____

**Calibrator Name:** _____

**Weather Conditions:** _____

**Mass Flowmeter Serial Number:** _____

**Port Number:** _____

| Observed Flow Rate (l/min) Rotameter | Actual Flow Rate (l/min) Mass Flowmeter | Time (min.) | Observed Volume (l) Digital Readout | Calculated Volume (l) Mass Flowmeter |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | Percentage Error: ± _____% | |
| | | | | |
| | | | | |
| **Action Required:** | | | | |
| **Action Taken:** | | | | |

factors so that the air sampling volumes may be corrected on the field submission sheets. Any air sampler which is calibrated and found to have a volume counter error of ±10% or more is to be immediately removed from the field. A corrective action form is to be filled out by the regional technician and sent to the QA Coordinator whenever an instrument is out of this acceptable calibration range.

3) Preventive Maintenance

Specific preventive maintenance checks and procedures are described in detail for the air sampling measurement systems in Section 4.5 of the Technical and Operating Manual and are summarized in Tables 4.12 and 4.13. These procedures must be followed on a regular basis to ensure sampler reliability and to meet the network objectives for data completeness (e.g. 5 day and 10 day criteria for event and cumulative networks respectively).

4.2.2.3 Sample Collection and Handling

The sample handling and collection procedures for the air sampling networks are much simpler than those of the precipitation sampling, and therefore have simpler QA/QC procedures. The major objective of the air filter sampling and collection procedures is to

TABLE 4.12

Air Sampling Preventive Maintenance - Daily Air Sampling

| Type of Service | Service Requirements | Service Frequency |
|---|---|---|
| Technician/Operator Checks | - clean filter housing shelter<br>- check timer, flow rate, pump<br>- ensure all parts are sequencing normally<br>- check "O" rings | weekly |
| Scheduled Maintenance | - inspect all internal tubing contacts and connections | every 2 months |
| | - replace in-line filters on sampling ports | every 6 months |
| | - overhaul pump (replace diaphragm, cover gasket, filter sponges, flaps) | every 6 months |
| | - replace sample line tubing and tighten boom couplings | annually |
| | - check all tubing and baseplate connections | monthly |

TABLE 4.13

Air Sampling Preventive Maintenance - Cumulative Air Sampling

| Type of Service | Service Requirements | Service Frequency |
|---|---|---|
| Operator Checks | - check flow rate, counters | weekly |
| Technician Checks | - check flow rate, volume and time counters<br>- check pump for noises<br>- check all external tubing<br>- check for leaks<br>- ensure sampling pole is secure and level<br>- adjust turnbuckles if necessary | monthly if possible |
| Scheduled Maintenance | - overhaul pump (replace diaphragm, cover gasket, filter sponge and flaps)<br>- check internal wiring and tighten connections<br>- replace sample inlet tubing<br>- replace in-line filter | annually (preferably semi-annually)<br>every 3 months<br>annually<br>every 3 months |

minimize potential sample contamination. To meet this objective, the system has been designed to reduce sample handling and to ensure that all samples are handled under controlled conditions.

In order to ensure that the documented procedures are being properly followed, operators (in the cumulative network) and regional technicians (in the event network) are to be audited internally by the QA Coordinator on a annual basis. The audit checks outlined in Section 4.4 are to be incorporated into these internal audit procedures. These audits are also to assess the data documentation aspects of the air sampling to ensure that sampler flow and time information are properly documented. These data documentation requirements have been described in detail in the Technical and Operating Manual. The internal audit by head office personnel is to include the inspection of the filter preparation facilities at the regional office.

Data summary sheets are to be used in air sampling networks to maintain a log of instrument status during sampling periods. In the cumulative air sampling network, the monthly summary sheet is combined with the cumulative precipitation monitoring summary sheet (Fig. 3.2). In the daily air sampling network, the form presented in Figure 4.12 is to be used by regional technicians and submitted to the QA Coordinator on a monthly basis.

FIGURE 4.12

Daily Air Sampling Summary Sheet

Month Year: _____ Site: _____

| Day | Instrument Status | Corrective Action Taken |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |
| 21 | | |
| 22 | | |
| 23 | | |
| 24 | | |
| 25 | | |
| 26 | | |
| 27 | | |
| 28 | | |
| 29 | | |
| 30 | | |
| 31 | | |
| QA/QC Samples | | |
| Handling Blanks Submitted: (circle) | Yes/No | |
| COMMENTS: | | |

Status Coder: M - malfunction
✓ - no problems

4.2.2.4 QA/QC Parameters for Air Sampling

The nature of the air filter samples limits the extent of QA/QC samples that can be obtained. For example, split or duplicate samples are not meaningful because the nature of filter loading of particulate material may not permit representative sample splitting, and controlled particulate loading experiments to check the accuracy of filter extractions and analyses are certainly not applicable on a routine basis. The sample quality control checks for air samples are therefore limited to 1) overall estimates of sampler precision through co-located sampling, 2) sample accuracy relative to sample contamination through the collection of dynamic filter handling blanks, and 3) filter blanks. The details of each of these types of QA/QC samples are described below and summarized in Table 4.14. Detailed procedures for collecting and submitting these types of samples are contained in the Technical and Operating Manual.

1) Co-located Sampling

One co-located daily air sampling system is currently operated at the Dorset site in order to obtain a measure of the precision of this type of measurement on a continuous basis. In the cumulative air sampling network, one co-located sampler is employed within each region.

TABLE 4.14

Summary of Routine QA/QC Procedures for Air Sampling

| QA/QC Sample or Procedure Type | Quality Assurance Parameter Measured | Frequency |
|---|---|---|
| Co-located Sampling | - overall site/sample precision | - continuous |
| Filter Handling Blanks | - sample accuracy relative to sample contamination | - quarterly |
| Filter Blanks | - procurement QA | - 5 from each new batch |

2) Filter Handling Blanks

In both the event and cumulative air sampling networks, a "filter handling blank" is to be submitted quarterly by regional technicians to investigate potential sample contamination. These handling blanks consist of filter packs that have been subjected to all the loading and handling procedures of the actual samples. These packs are to be left at a site for either a one week period (daily network) or a 28-day period (cumulative network). The cumulative handling blanks are to be rotated around sites in every region.

3) Filter Blanks

Five blank filters from each batch of filters prepared or purchased are to be submitted by the head office technician for chemical analysis prior to sending them to the field for use. In this manner, the initial blank filter quality is both assessed and assured.

4) QA/QC Data Reporting

The air sampling QA/QC data is to be reported in a manner similar to the precipitation data. In particular, the results of the co-located sampling and analysis of blank filter data are to be analyzed and reported by the QA Coordinator in the quality assurance reports, and discussed by network management during the annual review meetings.

# 5. LABORATORY SAMPLE ANALYSIS

## 5.1 Laboratory Sample Reception and Handling

The sample and data custody procedures followed throughout the network in field operations (Section 3.2), and within the laboratory, are extremely important from a quality assurance point of view. The complexity of the laboratory system leads to the involvement of many people in the handling of samples.

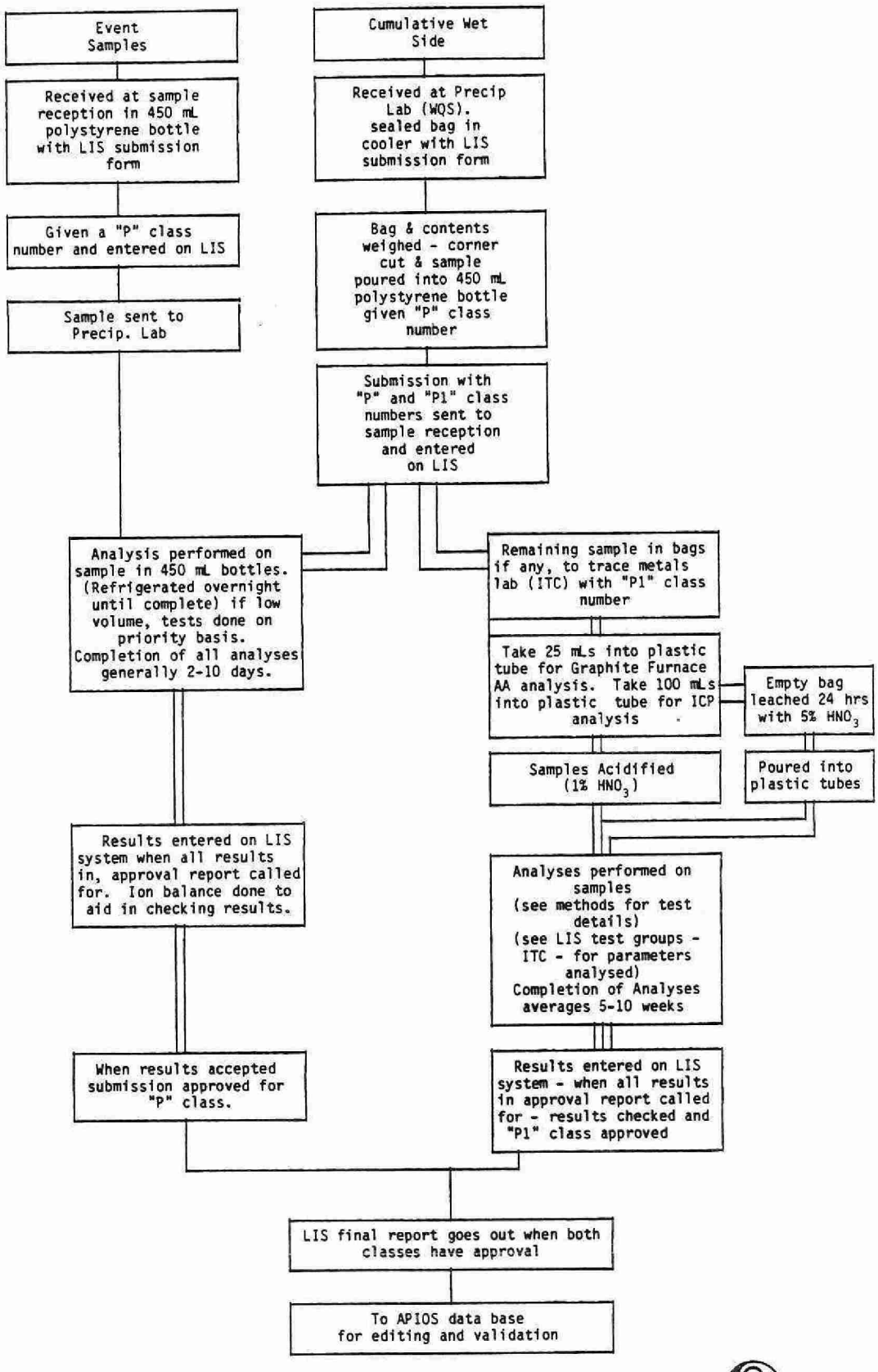
Specific sample custody and handling procedures have been developed for each sample type and are described below. These procedures ensure that storage conditions and times are appropriate for the types of samples being processed.

### 5.1.1 Precipitation Samples

The precipitation samples are shipped in insulated containers with cold packs to maintain low temperatures. Once delivered to the laboratory they are immediately placed into cold storage until they are processed at the sample reception desk. A flow diagram of the laboratory sample handling procedures for precipitation samples is provided in Figure 5.1.

FIGURE 5.1

Laboratory Sample Handling Procedures - Precipitation Samples



Event precipitation samples are delivered to sample reception in 450 mL polystyrene bottles along with their laboratory submission forms. The samples are assigned laboratory numbers, entered into the Laboratory Information System (L.I.S.) and sent to the precipitation laboratory for analysis. Samples are refrigerated at 4°C prior to analysis.

Sealed cumulative sample bags are delivered directly to a technician in the precipitation laboratory with laboratory submission forms. The bags and contents are weighed and the volumes are recorded on the laboratory submission form. The sample is then transferred into pre-labelled sample bottles. The transferring procedure involves washing one of the bottom corners of the bag, elevating this corner, cutting it off to form a small spout with clean scissors and transfering the sample. The sample bottle label is then checked again to ensure that no labelling errors have occurred. The bottles are placed in refrigerators immediately, kept at 4°C until sample analysis procedures are initiated.

The laboratory submission forms are sent to Sample Reception and entered in the L.I.S.

If samples of low volume (85 mL or less) are obtained, the analyses are carried out with the following priority volume, pH, acidity, $SO_4^{=}$, $NO_3^{-}$, $Cl^{-}$, $N\text{-}NH_4^{+}$, $Ca^{++}$, $Mg^{++}$, $Na^{+}$, $K^{+}$, and conductivity.

Once an analytical "run" has been identified the samples are redirected to the appropriate work stations for further analysis. Samples are to be removed from the refrigerator only when required for analysis. Care must be taken to ensure that samples sit at room temperature only when required, and that they are returned to the refrigerator immediately if analysis cannot be completed.

The analytical results are entered in the L.I.S. as each analysis is completed. When all analyses are entered, ionic balances are performed and results are screened and approved by the laboratory supervisor.

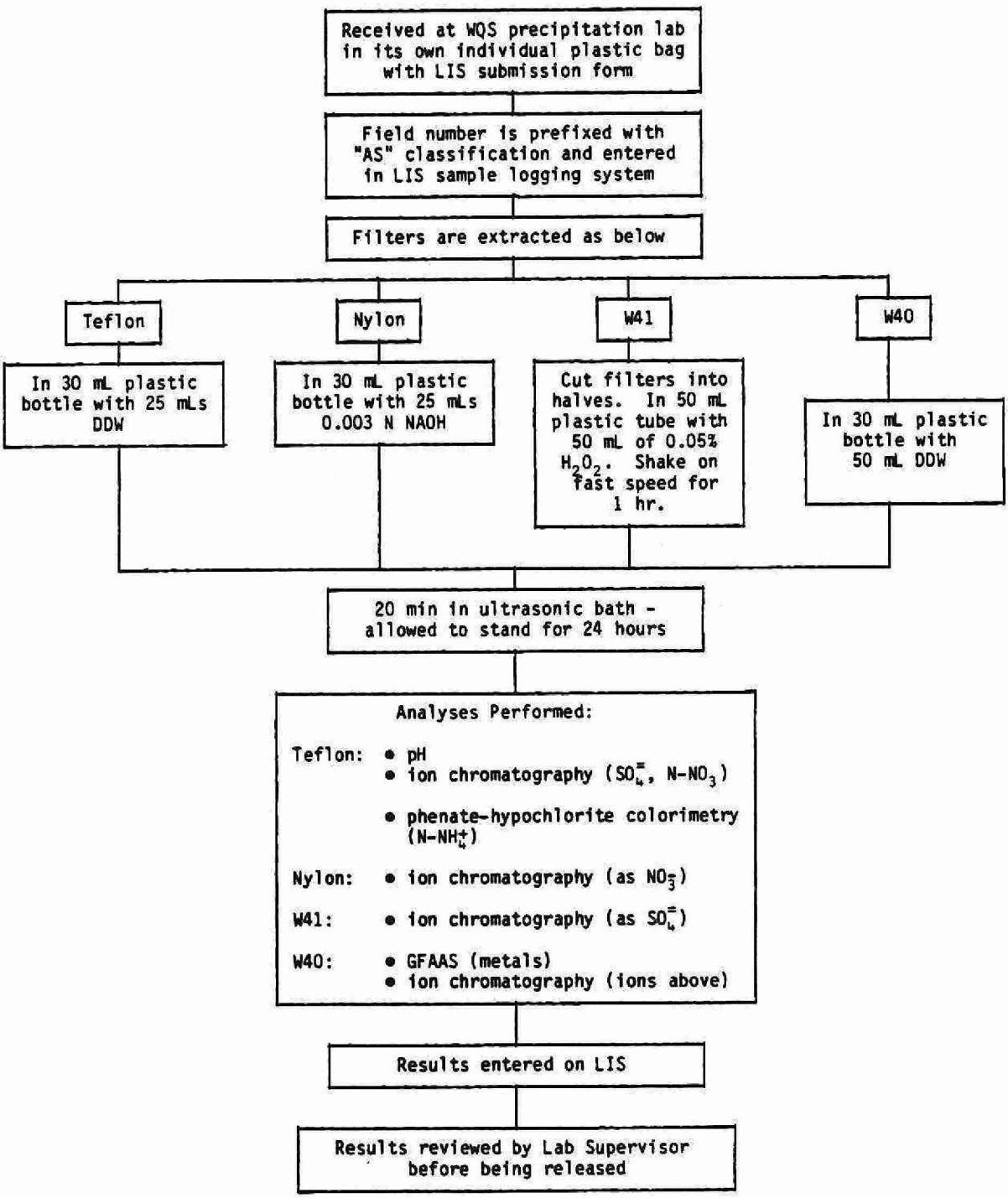
5.1.2 Air Filter Samples

Air filters and submission sheets are received in the precipitation laboratory by a technician. Identification data for the filters are recorded in a log. A flow diagram of the laboratory sample handling procedures is provided in Figure 5.2.

The filters are stored individually in labelled whirlpack bags at the laboratory while waiting for sample preparation and distribution. A pair of clean tweezers assigned to each filter type must be used while handling filters. These tweezers are to be cleaned with distilled dionized water and dried using kimwipes before handling each filter.

FIGURE 5.2

Laboratory Sample Handling Procedures - Air Sampling Filters



The analytical results are entered into the L.I.S. as each analysis is completed and when all results are entered they are reviewed by the laboratory supervisor before they are released. Analytical results for QC samples are entered in a performance report program.

## 5.2 Analytical Methods

The ultimate objective of the quality assurance and quality control procedures applied to the laboratory portion of the network is to ensure that the data produced by individual analysis methods are complete, of highest possible quality, and reported with information on accuracy and precision of analysis. It is also important to ensure that the chemical analysis results are obtained using sound techniques and proper laboratory procedures. This will facilitate use of the data for calculations of air concentrations and deposition fields and allow valid comparisons with other networks.

Analytical procedures used by the laboratory to analyze samples collected in the APIOS Deposition Program are documented in the "Handbook of Analytical Methods for Environmental Samples Volumes 1 and 2". Tables 5.1 to 5.4 list the laboratory analytical methods for parameters routinely measured in APIOS network samples. Changes in either analytical methodology or in the protocols used in an analytical method are to be immediately reported by memorandum to the network manager.

TABLE 5.1

Event Wet Deposition Network Laboratory Analysis

| Parameter | Analysis Method |
|---|---|
| Volume | Top loading balance |
| pH | Radiometer pH meter |
| Total acidity | Gran titration |
| | Potentiometric titration with pH meter |
| Conductivity | Conductivity meter |
| Chloride, sulfate, nitrate | Ion chromatography |
| Ammonium | Automatic colorimetry (phenate hypochlorite) |
| Calcium, magnesium, potassium, sodium | Flame atomic absorption |

TABLE 5.2

Cumulative Wet Deposition Network Laboratory Analysis

| Parameter | Analysis Method |
|---|---|
| Volume | Top loading balance |
| pH | Radiometer pH meter |
| Total acidity | Gran titration<br>Potentiometric tritration with pH meter |
| Conductivity | Conductivity meter |
| Chloride, sulfate, nitrate | Ion chromatography |
| Calcium, magnesium, potassium, sodium, zinc | Flame atomic absorption |
| Total Kjeldahl nitrogen<br>Total phosphorus<br>Ammonium | Automatic colorimetry |
| Iron, nickel, copper, lead aluminum, cadmium, vanadium | Graphite furnace atomic absorption |
| Manganese | Inductively coupled plasma |

TABLE 5.3

Daily Dry Deposition Network Laboratory Analysis

| Parameter | Filter Type | Analysis Method |
|---|---|---|
| Ammonium | Teflon | Automated colorimetry |
| Sulfate, nitrate | Teflon | Ion chromatography |
| Nitric acid | Nylon | Ion chromatography |
| Sulfur dioxide (as $SO_4^{=}$) | Impregnated Whatman 41 | Ion chromatography |

TABLE 5.4

Cumulative Dry Deposition Network Laboratory Analysis

| Parameter | Filter Type | Analysis Method |
|---|---|---|
| Zinc, iron, nickel, copper, lead, aluminum, cadmium, manganese, vanadium | Whatman 40 | Flameless AA |
| Ammonium | Whatman 40 | Automated colorimetry (phenate hypochlorite) |
| Calcium, sodium, potassium, magnesium | Whatman 40 | Flame atomic absorption |
| Nitrate, chloride, sulphate | Whatman 40 | Ion chromatography |
| Nitric Acid | Nylon | Ion chromatography |
| Sulfur dioxide (as $SO_4^=$) | Impregnated Whatman 41 | Ion chromatography |

A Quality Assurance Summary for each individual analytical method is found in Appendix III. These QA Summaries list:

- the sample type;
- preparation and handling procedures;
- instrumentation;
- operating characteristics; as well as
- specific QC procedures associated with the method.

These ensure that the method meets the network's requirements in terms of detection limits, accuracy, precision, and performance of the laboratory systems with respect to the expected processing time for network samples.

## 5.3 Laboratory QA/QC

The major objective of a laboratory quality control program for chemical analysis is to ensure immediate detection and solution of problems with the analytical procedures. These can include deterioration in instrumentation, chemical reagents and standards, techniques or calibration. A systematic application of quality control procedures of two major types are required to perform this function. Firstly, "function checks" in which the validity of the sample and performance of the equipment is controlled and secondly, "control checks" in which the

performance of the analytical system is verified and the results are monitored for acceptability in precision and accuracy.

5.3.1 Function Checks

The main objective of function checks is to confirm that the instrument operated correctly and that the analytical procedure ran under proper conditions at the time of analysis. These types of checks are to be carried out prior to and during each analytical run to confirm operational integrity.

Chemicals purchased for the quality control program are to be high purity materials and analyzed for contamination. Manufacturer and lot number of all chemicals used for standards are to be recorded and any change in manufacturer or lot number of the chemical should require checks of the quality. Standards are to be confirmed as accurate by comparison with standard reference materials and if possible, analyzed by another technique. Reagents are to be tested for contamination and composition. Distilled and deionized water sources are to be monitored for conductivity and specific blank levels. Transfer lines carrying these supplies are to be monitored for algae and dirt build up and replaced when necessary. Sample containers, filters and any other equipment used must be checked for contamination, leaching and adsorption.

All glassware used in the laboratory is to be thoroughly cleaned and rinsed prior to use. Any instrument used in an analysis must be tested to ensure that it is functioning according to specifications. For example, zero and full-scale readings are to be properly adjusted, the instrument should not be influenced by stray signals such as line voltage transients and should not show any sudden shifts. The instrument is to be kept clean, in proper working order and warmed up according to specifications before use. Cuvets and flow cells used in colorimetric tests should be cleaned with appropriate reagents and techniques. Electrodes should respond rapidly and hold a steady response or else be replaced. All band pass filters are to be scanned to ensure that the proper wavelengths are transmitted and that the band width is as specified. Instruments that use multiple scales, such as conductivity and pH meters are to be checked for interscale comparability. Spectrophotometers are to be checked to ensure accuracy in wavelength calibrations. In short, function checks are to include the following procedures:

- verification of sample integrity including expiry date and storage or sample handling procedures (via sample and data custody procedures);

- checks on instrument stability, drift, warm-up time, etc. prior to operation;

- checks that zero, span and operating ranges are properly adjusted;

- check that operation is under controlled environmental conditions (particularly temperature).

Many of the checks performed as part of "function checks" consist of good laboratory practice procedures which are well documented in appropriate laboratory manuals (MOE, 1983e). It is important that analysts be thoroughly trained (Table 3.2) and familiar with the instruments and procedures.

5.3.2 Control Checks

The major goals of the control checks applied to the analytical procedures are to provide a measure of the accuracy and precision associated with the data, and to identify deteriorating or "out of control" conditions as they develop. This ensures that immediate attention is given to analytical problems. These control checks consist of many different types of samples (blanks, spike or real samples) and are discussed below.

5.3.2.1 Calibration Procedure

Instrument calibration is an operating procedure performed to identify the instrumental response to known concentrations of relevant compounds. This allows the instrument response to the corresponding concentration of "unknown" sample to be related to the concentrations of the compounds. Calibration consists of preparation and analysis of standard solutions (traceable to reference materials) over the full range of instrument responses. Calibration curves determined by these standards provide the relationship between sample concentration and instrument response.

A gradual change, either increase or decrease in peak height of low and high standards, may be due to a systematic change in analytical conditions. Sudden shifts in sensitivity should be investigated immediately. The baseline should not shift more than 5% of full scale over the course of a run. Sensitivity shifts in pH and conductivity meters should be detected and corrected at regular intervals.

The following steps are taken when setting up most of the analytical instruments.

1. Deionized water (or another appropriate "blank") is analyzed to adjust the baseline or zero setting of the instrument to a suitable region on the chart recorder or other instrument readout.

2. If the system is known to be linear and stable, a high standard at 80% or 100% of full scale is analyzed a number of times to check instrument sensitivity. The system is not calibrated until the second standard is run to ensure analytical precision and accuracy and to avoid bias due to contamination or other effects. Subsequent standards may be adjusted to the proper reading by means of a calibration or gain adjustment. These changes should be made only if the quality control standards are not within acceptable ranges.

3. When the blank and high standard have been set, a low standard at about 10% - 20% of full scale is analyzed. If it reads incorrectly, this may indicate curvature of the calibration, contamination of the blank water, or improper preparation of either of the two standards. The cause of the problem must be corrected or documented.

4. If the system is known to have a non-linear calibration, several standard analyses are required. A daily calibration curve can be drawn from this data.

5.3.2.2 Calibration Verification

The calibration of each analytical procedure is verified by means of two control standards QC-A and QC-B which are made up indepen-

dently of the calibration standards and are chosen to be about 70% and 10% of full scale, respectively.

When the QC-A and QC-B control standards are analyzed and read, their sum and difference are calculated, plotted for each run on a control chart and used immediately by the technician to determine whether the calibration process is in control. This involves the concepts of Warning, Control and Acceptance Limits as defined by variation in the difference (D) between QC-A and and QC-B standards. This is estimated by the standard deviation of the differences ($S_D$) from previous data and is a measure of within-run variation.

Warning Limit: If the current value of QC-A - QC-B differs from the defined value by more than 2 $S_D$, then recent past values should be reviewed to determine if a discernible trend is developing. Analysis may proceed if this is not apparent. In general, the warning limits are not expected to be exceeded more than once in twenty times.

If the current value of QC-A + QC-B differs from the defined value by more than 2 $S_D$, then systematic calibration error may be present. The recent trend in the plot of QC-A + QC-B should be examined carefully.

Control Limit: If the current value of QC-A - QC-B differs from the defined value by more than 3 $S_D$, then today's calibration slope is out-of-control. Since these limits are not expected to be exceeded more than 1 time in 200, there is probably something wrong with the system. The analytical run must be stopped until a cause is determined. The system must be recalibrated and rechecked using QC-A and QC-B. On the short-term, if the slope is in control, control limits for QC-A + QC-B should be set at 3 $S_D$ to prevent significant "blank correction" bias where this is important.

Acceptance Limit: Systematic error in estimating slope and blank corrections will cause the sum (T) of QC-A and QC-B to vary excessively. Over the long-term, in order to control between-run standard deviation, the standard deviation ($S_T$) of QC-A + QC-B should not exceed $S_D$ by a factor of more than 1.5. Therefore if QC-A + QC-B exceeds the Acceptance Limit of 4.5 $S_D$, the presence of excessive systematic error is suspected. The analytical run is stopped and the system is recalibrated.

Controls such as standard calibration sensitivity, cell constant or gain are to be monitored for each analytical run. Any large change in these settings should be viewed with suspicion if all standards give the proper readings. If QC-A - QC-B is in control but QC-A + QC-B is out of control, a blank or baseline correction may be required. If both QC-A - QC-B and QC-A + QC-B are out-of-control, an analytical problem exists, either one or both of A and B may be in gross error, reagents may be improperly prepared or the analytical instrument may be experiencing a problem.

The actual values of QC-A and QC-B are examined to see if either is suspect whenever their difference or sum is found to be out-of-control, but they are not individually out-of-control. However, over the long-term the standard deviations of QC-A and QC-B ($S_A$ and $S_B$, respectively) can be used to estimate the between-run standard deviation and its dependence on concentration. In some systems, the in-run standard deviation is not particularly concentration dependent, although the between-run standard deviation may be. The concept of the Acceptance Limit recognizes that data users do not always require the ultimate performance as estimated by the variation of in-run duplicates.

Drift in the control standards, the sum (QC-A + QC-B) and the difference (QC-A - QC-B) are to be monitored by plotting concentration

values against run on a control chart. Technicians are to plot their control data on these charts daily, as well as keeping all the data in a log book.

In practice, the Acceptance Limit for Sums and the Control and Warning Limits for Differences may be made more stringent to achieve a particular benefit, but, in general, they should not be set much more stringent than the system can tolerate, and should never be set looser than what long-term performance data suggests can be maintained routinely.

5.3.2.3 Recovery Checks

In methods requiring sample preparation such as digestion or extraction, a recovery check suitable to that system is necessary to estimate the efficiency of the preparation method. These solutions are not used to calibrate the instrument, but corrections for the digestion blank and matrix effects can be estimated and applied if necessary. Recovery standards are chosen to test all facets of the analysis. If a digestion step results in extraction of a substance or conversion of one substance into another before analysis, the recovery standard should be chosen to test the efficiency of this step. For example, glycine is used to test the efficiency of the digestion in the total Kjeldahl nitrogen test. Without digestion, glycine might give a zero response.

Duplicate recovery standards are to be prepared at 0%, 20%, 40% and 80% of full scale. These recovery standards are analyzed in the same manner as routine samples and results are compared to the untreated calibration standards. The recovery efficiency is reported as a percent recovery and the recovery step should have a high efficiency. Usually this is set greater than 95 %.

Where a number of these recovery standards have been read, a mean and standard deviation are calculated for each. If $R_1$ and $R_2$ are 80% and 40% standards respectively then in order for a run to be judged valid the following conditions should be met, for example:

1. the average values of $R_1$ and $R_2$ are to be greater than 95% of their theoretical values;

2. the day's values of $R_1$ and $R_2$ are to be within 3 standard deviations of their means;

3. the day's blanks are to be within 3 standard deviations of their mean.

If both $R_1$ values are out, the run must be repeated. If one $R_1$ and one $R_2$ are within the 3 standard deviation limits, then the run may proceed with approval of the senior technician after affirmation that systematic problems do not exist with the method.

Poor agreement between duplicate standards indicates deterioration of within-run precision or problems with recovery of the recovery check standards.

5.3.2.4 Precision

Precision refers to the reproducibility of a measurement technique when it is repeated on a homogeneous sample under controlled conditions.

Natural samples are selected on a random basis and run in different parts of the run to assess precision. Generally one sample in twenty up to a maximum of 3 per day is run in duplicate. One sample of each pair is analyzed at the beginning of the run with the QC standard and recovery standard. The other sample of the pair is analyzed in the regular sample order. Duplicates are segregated into 3 ranges which are expressed as percentage of full scale, these are: 0-20%, 20-50% and 50-100% for evaluation of precision.

Standard deviation values from within-run duplicates are to be used to estimate the within run precision, detection criteria and detection limit. (Detection criteria and detection limits are derived from the within-run precision of low level samples and are defined as 1.64 times and 3.28 times the within-run precision respectively, at a 95 % confidence level).

5.3.2.5 Accuracy

Accuracy refers to the agreement between the average of several measurements and the accepted or "true" value. Accuracy is generally expressed as the difference between these two values or as relative error where the difference is expressed as a percentage of the accepted value. An estimate of the laboratory's accuracy is obtained from intercomparison studies with other laboratories or by analyzing standard reference materials (SRM). The SRM's are available from organizations such as USEPA in Cincinnati or from the US National Bureau of Standards. These SRM's are used to test the laboratory's ability to analyze pure standards and to establish bias for the preparation and analysis protocols.

Intercomparison studies sometimes have pure standards, however, they often contain some stabilized real samples. Successful analysis of a pure standard but failure to obtain comparable results for real samples even when all the quality control objectives have been met

indicate that the analytical technique used may lack specificity for the parameter or may be subject to an interference from the sample matrix. Analysis of real samples may also indicate difficulties in the preparation or recovery procedures.

The Water Quality Section and Inorganic Trace Contaminant Section are to participate in relevant interlaboratory comparison studies at least twice every year. The results of these comparisons are to be reported to the QA Coordinator and Network Manager of for the APIOS network.

5.3.3 Data Screening

While the quality control steps outlined here are helpful to the analyst at the bench, the real utility arises from a review of the data to assess performance of the analytical systems at regular intervals.

Therefore, before data results are reported to the network, some preliminary data screening and validation including assessment of QA/QC data is to take place. The senior technicians should screen all data sets and their associated QA/QC data to ensure that all requested analyses were undertaken, that proper QA/QC procedures were performed,

that data transcription errors are absent and that unreasonable or obvious errors are investigated.

In addition, a number of data screening procedures or "consistency checks" are to be applied before the data is released. These are described below.

a) Ion Balance - consisting of a simple calculation of the ion balance in which the ratio of cations and anions is determined. For results on a sample to be judged acceptable, this balance should be within 10 % for precipitation samples. The more ions which are analyzed the easier it should be to achieve balance between anion and cation equivalents.

b) Calculated pH versus measured pH and/or measured vs theoretical conductivity should also be within acceptable limits.

c) Relationship between total Kjeldahl nitrogen and ammonia should always show greater or equal amounts of total Kjeldahl nitrogen compared with ammonia.

If these screening procedures are carried out immediately after the analyses have been completed, it is often possible to catch errors or omissions by reanalyzing the sample before samples are discarded.

Tables 5.5 to 5.9 summarize the various types of control samples discussed in this section. These summaries list the type of sample, a brief description of each, the nature of the control and the frequency of its use.

A schedule for the laboratory quality control activity and level of effort is summarized in Tables 5.10 to 5.14.

TABLE 5.5

Summary of Laboratory and Network Quality Control Sample Types - Laboratory Blanks

| Type of Sample | Description | Nature of Control | Frequency |
|---|---|---|---|
| Blank | Deionized water (or another appropriate "blank") is analyzed to adjust the baseline or zero setting of the instrument to a suitable reading on the chart recorder or other readout. | Set up analytical instrument. Detection of deterioration in instrumentation. | Daily |
| Reagent Blank | Deionized water QC sample is subjected to the same preparation procedure as routine samples to be analyzed. | To check for random contamination which may occur in sample preparation or reagents. A measure of lab bias. | Anytime there is a change in manufacturer or lot number |
| Handling Blank | Deionized water QC sample is subjected to the same handling procedures as routine samples being analyzed. | To check for random contamination leaching & adsorption which may occur in sample handling, i.e. glass ware, sample containers, bags, etc. A measure of lab bias. | Quarterly |
| Filter Blank | Filters are subjected to the same preparation procedures as routine samples to be analyzed. | To check for random contamination which may occur in sample preparation or filters. A measure of filter contamination. | Anytime there is a change in filter lot numbers |

TABLE 5.6

Summary of Laboratory and Network Quality Control Sample Types - Field Blanks

| Type of Sample | Description | Nature of Control | Frequency |
|---|---|---|---|
| Dynamic Blank | a) Deionized water QC sample is poured into sample container and subjected to the same handling procedures as routine samples being analyzed. | To check sampling containers and handling procedures for possible contamination. | Quarterly |
| | b) Composite QC sample is poured into sample container at various times subjected to the same handling procedures as routine samples being analyzed. | To check field and lab bias. | Quarterly |
| | c) Filter pack loaded, left at sampling site, unloaded then subjected to the sample handling procedures as routine samples being analyzed. | To check field and lab bias. | Quarterly |
| Blank Bag/Bottle | Check new inventory of bottle and bags for contamination. | To investigate the potential contamination or sorption characteristic. | When procuring new bags or bottles |

TABLE 5.7

Summary of Laboratory and Network Quality Control Sample Types - Re-Analysis of Real Precipitation Samples

| Type of Sample | Description | Nature of Control | Frequency |
|---|---|---|---|
| Different Run | Randomly chosen previously analyzed samples. | Provides information on analytical precision for different days of analysis.<br><br>CHECK - between run repeatability. | Daily |
| Within Run | Randomly chosen previously analyzed sample within the same run. One is analyzed at the beginning of the run with the QC standard and recovery standard and the other of the pair is analyzed in the regular sample order. | Provides information on analytical precision within a run. Analyst can use standard deviation values from within run duplicates to estimate the within run precision, detection criteria and detection limits. | Daily |
| Split Samples | Randomly chosen precipitation samples split into two aliquots by the QC chemist and submitted "blind" to the laboratory for analysis. | To check within lab precision. | Monthly |
| Co-located Field Samples | Samples submitted to laboratory from co-located sites. | To check operational, lab and network precision. | - 1 site per region/network<br>- daily air network 1 site |

TABLE 5.8

Summary of Laboratory and Network Quality Control Sample Types - Prepared and Standard Sample

| Type of Sample | Description | Nature of Control | Frequency |
|---|---|---|---|
| Calibration Standards | Prepared by analyst.<br><br>Standard solutions covering the range of instrument responses are analyzed before the analytical run commences. | To check within run accuracy, laboratory bias and instrument response. | Daily |
| Quality Control Standards | Prepared by analyst.<br><br>Two standard solutions QC-A, QC-B are made up and maintained independently of the calibration standards at about 70 and 10% of full scale. | To check within and between run accuracy used as calibration control, control charting and Youden plots - to control slope and blank bias. | Daily |
| Internal Lab Audit Samples | Prepared by Quality Control Chemist which are made up and maintained independently of the calibration standards and submitted "blind" to analyst. | To check analytical accuracy and assess data quality independently of the laboratory analyst. | Monthly |
| Spiked Real Samples | Spiked sample prepared by the Quality Control Chemist submitted "blind" to the analyst.<br><br>Where spiked sample = simulated rain water samples containing all major constituents.<br><br>QC chemist calculates % recovery. | To check sample recovery. | Monthly |
| Standard Reference Samples | Analysis of standard reference materials. | To establish bias for the calibration and analysis steps. | Annually |
| Recovery Standards | Recovery standards prepared at 0%, 20%, 40%, and 80% of full scale. Analyzed in the same manner as regular samples. | Test efficiency of digestion.<br><br>Correct for digestion blank and matrix effect.<br><br>Calculate % recovery. | Daily |

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TABLE 5.9

Summary of Laboratory and Network Quality Control Sample Types - External QA/QC Activities

| Type of Sample | Description | Nature of Control | Frequency |
|---|---|---|---|
| Round Robins<br>Standards<br><br>Audits<br><br>Prepared<br>Synthetic<br>Samples | QC samples submitted with the knowledge of the laboratory but "blind" to the analyst and QC samples submitted "blind" to the laboratory. | Measure lab accuracy. | Yearly |

TABLE 5.10

Quality Control/Quality Assurance Schedule

Daily Checks

| Activity | Analyst (Technician) | Senior Technician | Lab. Supervisor | QA Coordinator |
|---|---|---|---|---|
| QC-A/QC-B | ✓ | | | |
| Control Charts | ✓ | | | |
| Recoveries | ✓ | | | |
| Duplicates | ✓ | | | |
| Blank Charts | ✓ | | | |
| Visual Instrument Check | ✓ | | | |
| Make Necessary Changes | ✓ | ✓ | | |
| Instrument Check-zero and 100 % | ✓ | | | |
| Distilled Deionized Water Check | ✓ | ✓ | | |
| Approval QC-A; QC-B | | ✓ | | |
| Check Final Data | | ✓ | | |

TABLE 5.11

Quality Control/Quality Assurance Schedule

Weekly Checks

| Activity | Analyst (Technician) | Senior Technician | Lab. Supervisor | QA Coordinator |
|---|---|---|---|---|
| Review Control Charts | | ✓ | | |
| Review Recoveries | | ✓ | | |
| Make Necessary Changes | ✓ | ✓ | | |
| Check Standard Concentration by Other Techniques (e.g. Dionex for $Cl^-$ $NO_3^-$, $SO_4^=$ ) | ✓ | ✓ | ✓ | |

TABLE 5.12

Quality Control/Quality Assurance Schedule

Monthly Checks

| Activity | Analyst (Technician) | Senior Technician | Lab. Supervisor | QA Coordinator |
|---|---|---|---|---|
| Review Monthly Data | | ✓ | ✓ | |
| Make Necessary Changes | ✓ | ✓ | ✓ | |

TABLE 5.13

Quality Control/Quality Assurance Schedule

Annual Checks

| Activity | Analyst (Technician) | Senior Technician | Lab. Supervisor | QA Coordinator |
|---|---|---|---|---|
| Container Check | ✓ | ✓ | ✓ | ✓ |
| Filter Check | ✓ | ✓ | ✓ | ✓ |
| QA Report | | | | ✓ |

TABLE 5.14

Quality Control/Quality Assurance Schedule

As Required

| Activity | Analyst (Technician) | Senior Technician | Lab. Supervisor | QA Coordinator |
|---|---|---|---|---|
| New Reagent Checks | ✓ | ✓ | ✓ | ✓ |
| Inter Lab Round Robins | ✓ | ✓ | ✓ | ✓ |
| Field "Blind" Duplicates | ✓ | | | |
| Lab "Blind" Duplicates | ✓ | ✓ | ✓ | ✓ |
| Lab "Blind" Standards | ✓ | ✓ | ✓ | ✓ |
| Check New Cleaning Reagents | ✓ | ✓ | ✓ | ✓ |

# 6. DATA OPERATIONS

## 6.1 Data Management and Objectives

The overall objectives of data management are to collect, process, validate and report network data in a timely and efficient manner. In order to meet the general operating objective, specific quality assurance procedures are to be implemented to achieve the following:

1) ensure data collecting and processing procedures are well designed and implemented for all aspects of the network;
2) ensure current data validation procedures and software packages are documented and followed;
3) ensure that QA/QC systems are in place to monitor the effectiveness of the operating systems;
4) ensure that data are reported in a timely manner;
5) ensure that all reported data are accompanied by relevant QA/QC data.

The general QA/QC objectives have been stressed throughout the development of the APIOS data handling system. The details of the data operations and their relevant QA/QC procedures are discussed below in

terms of (1) data collection, (2) data screening and validation, and (3) data reporting. The general network data handling procedures that are to be followed are summarized in Figure 6.1

## 6.2 Data Collection and Entry

The QA/QC checks that are to be carried out on the raw data collected may be classified into two major types: (1) field data checks, and (2) head office data checks. The QA/QC checks for each type are presented below.

FIELD DATA COLLECTION CHECKS

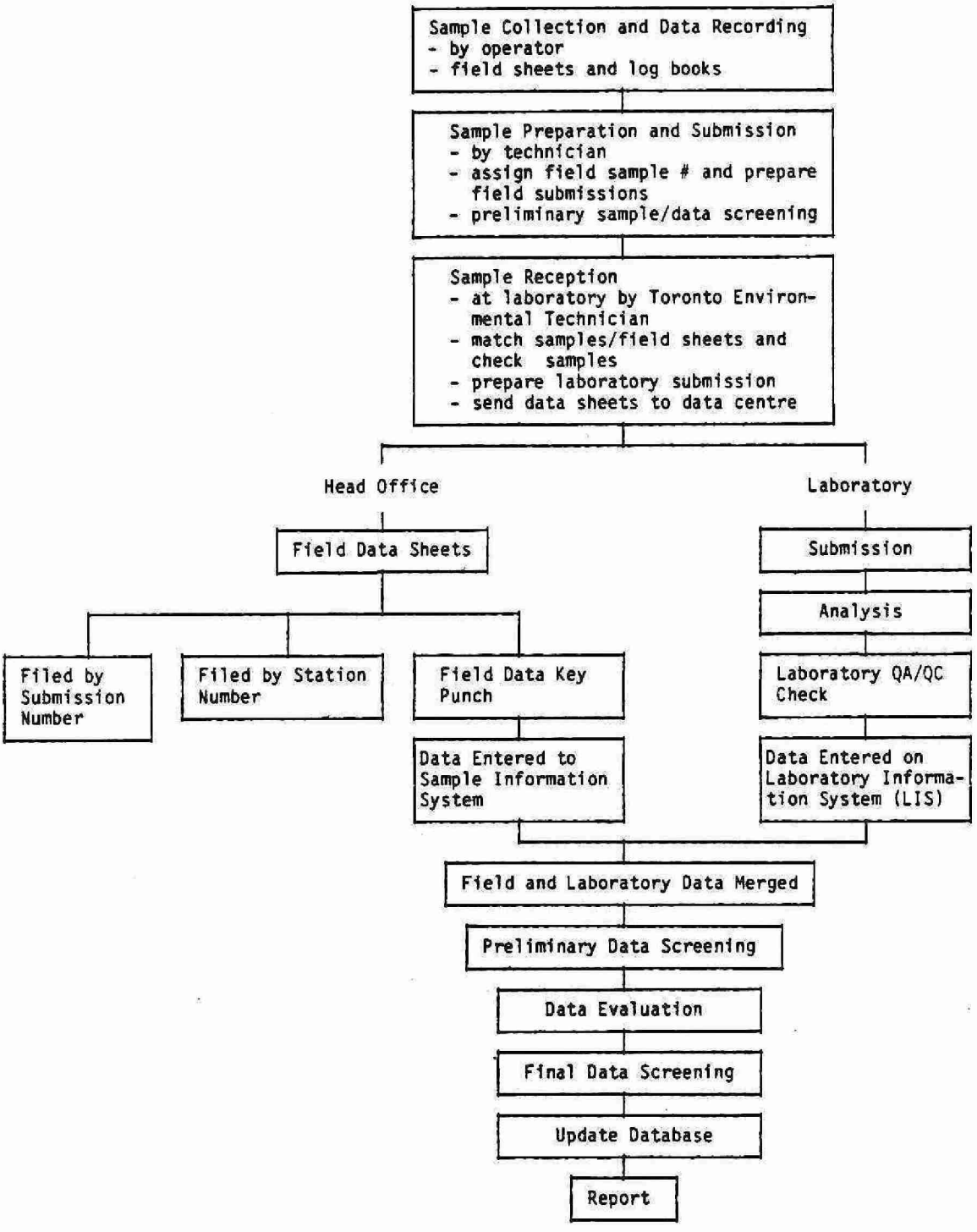
- Technicians ensure that all information is reported on operator sheets and log books before preparing field submission sheets.
- Technicians ensure QA/QC samples are submitted as required.
- Technicians prepare monthly field data summaries.

HEAD OFFICE DATA COLLECTION CHECKS

- Ensure all sample/data sheets match before submission to the laboratory (Toronto Environmental Technician).

FIGURE 6.1

Generalized Network Data Handling Procedures



- Check accuracy and completion of field data sheets and make corrections if required (Data Clerk).
- Check data merging by comparing laboratory (LIS) and office (SIS) data listing (Database Scientist).
- Ensure that all technician field data summaries have been properly prepared (QAC).
- Extract QA/QC sampling information (Database Scientist).

Once these generalized data checks have been carried out at the field and head office levels, the preliminary data set which is complete (in terms of data submitted) and accurate (in terms of sampling dates, labelling, etc.) is ready for further data screening and validation.

## 6.3 Data Screening and Validation

The network data screening and evaluation procedures refer to those QA/QC steps carried out to ensure the final quality of the data reported by the network. Several types of screening and validation techniques are used.

### 6.3.1 Preliminary Data Screening

The objective of the preliminary data screening is to identify any and all questionable data points for further investigation by a

network staff scientist. These checks include manual screening techniques as well as computerized checks including checks of sample integrity, composition and statistical analysis.

The manual data checks consist of "common sense" checks to ensure that all relevant data are included, completing any missing data, correcting obvious sample identification errors, ensuring all comment codes are included and correcting any keypunching errors. Once these "gross sample checks" have been completed, the data are subjected to a number of computer screening programs to create a listing of suspect data points to which data validation flags or sample comment codes are appended at the discretion of the staff scientist responsible for data evaluation. The specific details of the data screening procedures carried out during the computer screening are described below.

6.3.1.1 Computerized Data Screening for Precipitation Samples

Precipitation samples from both networks are validated utilizing three different techniques described below.

1. Sample Analysis Integrity Checks

The first approach applied to data validation is to check the integrity of sample analysis to determine the completeness of analysis

and to determine possible inaccuracies in analytical results. Integrity checks include ionic balance, theoretical vs. measured conductance, theoretical vs. measured pH, total hydrogen vs. free hydrogen and measured field pH vs. measured lab pH. Also, sample representativeness is determined by calculating sampler collection efficiency.

In the event precipitation network, the limits of % discrepancy for integrity checks are to be based upon the approximate 2.5 and 97.5 percentiles of cumulative distributions from the current year of data.

In the cumulative precipitation network, the discrepancy limits are to be based upon the 2.5 and 97.5 percentiles of the cumulative distribution of the daily precipitation data of the same period.

Ionic Balance (All concentrations are in mg $L^{-1}$):

anion equivalents =

$$\frac{(SO_4^{=}) \times 2}{96.1} + \frac{(NO_3^{-}\text{-}N)}{14} + \frac{(Cl^{-})}{35.5} + 0.001 \left[\frac{4.90}{10^{-pH} + 6}\right] \text{(bicarbonate correction)}$$

cation equivalents =

$$10^{-pH+3} + \frac{(NH_4^{+}\text{-}N)}{14} + \frac{(K^{+})}{39.1} + \frac{(Ca^{++}) \times 2}{40.1} + \frac{(Mg^{++}) \times 2}{24.3} + \frac{(Na^{+})}{23}$$

$$\% \text{ Discrepancy} = \frac{(\text{anion equivalence} - \text{cation equivalence})}{(\text{anion equivalence} + \text{cation equivalence})} \times 200\%$$

Theoretical vs. Observed Conductance Balance (all concentrations are in mg $L^{-1}$):

Conductivity (umho $cm^{-1}$) at 25° per mg $L^{-1}$:

| | |
|---|---|
| $Ca^{++}$ - 2.60[a] | $SO_4^{=}$ - 1.54[a] |
| $Cl^{-}$ - 2.14[a] | $NH_4^{+}$-N - 5.32[b] |
| $Mg^{++}$ - 3.82[a] | $NO_3^{-}$-N - 5.09[a] |
| $K^{+}$ - 1.84[a] | $H^{+}$ - 350[b] |
| $Na^{+}$ - 2.13[a] | |

[a] - from "Standard Methods for the Examination of Water and Wastewater" 14th Edition, 1975.

[b] - calculated from equivalent conductance - CRC Handbook of Chemistry and Physics.

Theoretical Conductance =

$$10^{-pH+3} \times 350 + (SO_4^{=}) \times 1.54 + (NO_3^{-}\text{-N}) \times 5.09 + (Cl^{-}) \times 2.14 +$$

$$(NH_4^{+}\text{-N}) \times 5.32 + (K^{+}) \times 1.84 + (Ca^{++}) \times 2.60 + (Na^{+}) \times 2.13 + (Mg^{++}) \times$$

$$\% \text{ Discrepancy} = \frac{(\text{Observed Conductance} - \text{Theoretical Conductance})}{(\text{Observed Conductance} + \text{Theoretical Conductance})} \times 200\%$$

Theoretical pH vs. Observed pH (all concentrations are in ueq $L^{-1}$):

As discussed by Stensland (1978), rain or melted snow maintains charge balance and if it is assumed that falling raindrops are in equilibrium with atmospheric carbon dioxide ($P_xCO_2 = 320 \times 10^{-6}$ atm) then the following equation can be proposed for the calculation of theoretical pH.

$$\text{Theoretical pH} = 6 - \log_{10}\left(((\text{Net Ions}) + ((\text{Net Ions})^2 + (4K_HK_1PCO_2))^{0.5})/2\right)$$

$$\text{Net Ions} = (SO_4^{=}) + (NO_3^{-}) + (Cl^{-}) - (Ca^{++}) - (Mg^{++}) - (NH_4^{+}) - (Na^{+}) - (K^{+})$$

$$K_H = 0.034 \times 10^{6} \text{ ueq/L/atm}$$

$$P_xCO_2 = 320 \times 10^{-6} \text{ atm}$$

$$K_1 = 4.5 \times 10^{-1} \text{ueq/L}$$

$$\%\ \text{Discrepancy} = \frac{(\text{Observed Lab pH} - \text{Theoretical Lab pH})}{(\text{Observed Lab pH} + \text{Theoretical Lab pH})} \times 200\%$$

Free Hydrogen vs. Total Hydrogen:

$$\text{Free } (H^{+}) = 10^{-pH+3} = \text{mg } H^{+}L^{-1}$$

$$\text{Total } (H^{+}) = \text{Total Acidity}/50 = \text{mg } H^{+}L^{-1}$$

$$\text{Ratio} = \frac{\text{Free } (H^{+})}{\text{Total } (H^{+})} = \frac{10^{-pH+3} \times 50}{\text{Total Acidity}}$$

Total acidity results are expressed in $CaCO_3$ equivalents ($mg\ L^{-1}$) of a titration of the sample with NaOH to an end-point pH of 8.3. If the ratio is found to be greater than 1, the office comment code "T" is appended to the record.

Observed Field pH vs. Observed Lab pH (event samples only):

$$\% \text{ Discrepancy} = \frac{(\text{Observed Lab pH} - \text{Observed Field pH})}{(\text{Observed Lab pH} + \text{Observed Field pH}} \times 200\%$$

Sample Representativeness or Collection Efficiency:

Calculation of Equivalent Precipitation Depth (mm)

Sangamo and M.I.C. Type Instrumentation:

$$\text{Equivalent Precipitation Depth (mm)} = \frac{\text{Volume Collected (mL)}}{1000} \times 30.8$$

Aerochem Metrics Instrumentation:

$$\text{Equivalent Precipitation Depth (mm)} = \frac{\text{Volume Collected (mL)}}{1000} \times 15.6$$

$$\% \text{ Efficiency} = \frac{\text{Equivalent Precipitation Depth (mm)}}{\text{Gauge Depth (mm)}} \times 100\%$$

The upper and lower limits for collection efficiency are determined subjectively to be 120.0% and 50.0% respectively. Reported collection efficiencies less than 50.0% may indicate missed events due to instrument malfunction or may indicate loss of sample volume due to evaporation. Collection efficiencies which exceed 120% may indicate invalid "Gauge Depth" values. Violation of the above limits results in the incorporation of the office comment code "N" into the database.

2. Gross Limit Checks

The second approach for data validation is to apply gross limit checks to identify unlikely values. Lower limits are generally defined as detection limits and upper limits are determined from cumulative frequency distributions based on current data. If a gross limit check is violated, a report is generated and the sample investigated further. The approximate 97.5 percentile is utilized for the upper limit and the 2.5 percentile or "detection limit" is utilized as the lower limit.

3. Statistical Check for Outliers

The third approach is to screen the data for outliers statistically by applying the Dixon Ratio test to the highest and lowest

values observed in each sample cluster or region on a daily or 28-day basis. Outliers are determined at the 95% level of confidence. If an outlier is detected the sample is investigated further.

6.3.1.2 Computerized Data Screening for Daily Air Sampling

The first approach utilized in data validation is to apply gross limit checks to identify unlikely values and to apply integrity checks to check sample flow volumes and sampling dates. The second approach is to examine the sample structure by principal components analysis. Sample records are qualified by three methods. Field comments are appended to sample records at points of collection to reflect sample quality and possible sources of contamination. Office comment codes are appended to sample records to indicate failure of specific integrity tests or of sample representativeness. If outliers are detected or gross limit checks fail, validation flags may be appended to individual analytical results.

The data validation process incorporates two editing/screening stages. The first stage is an editing process wherein the database output is compared with historical records to ensure completeness and accuracy of the dataset. The second stage is the subjective review wherein statistical procedures such as gross limits and principal compo-

nents are used to select unlikely analytical results. Unreasonable results are either corrected (if typographical) or flagged as unreliable. An analytical result flagged as unreliable may indicate any of the following: the reported result appears to be in error and does not represent the true sample; the reported result appears significantly influenced by contamination (wind blown particulates, etc.), or the reported result appears to reflect local sources, contrary to the network's objectives. No results are deleted from the database.

## Gross Limit Checks and Integrity Checks

Upper and lower gross limits are determined for each measured variable, by station and by season. The winter season includes November to April while the summer season includes May to October. Upper limits are set as M + 2S where median (M) and scale (S) represent robust estimates of mean and standard deviation respectively. The scale of the distribution is estimated from interquartile distance, i.e. S = 0.74 (3rd Quartile - 1st Quartile) based upon logarithmically transformed results. In a situation where the distribution is significantly bounded by reported detection limits, S may be estimated as follows, S = 1.48 (3rd Quartile - 2nd Quartile). All lower gross limits are to be specified as adopted detection limit values. If a gross limit is exceeded, a report is generated and the sample is listed for further investigation.

Integrity checks consist of checking flow volumes and sample dates. Any sample which has a total flow volume of less than 10,000 litres per day is appended with the code "F", while any abnormal sampling period (> 1 day) is appended with the code "Z".

Sample Structure Analysis

The sample structure, as represented by the analytical results, is examined for outliers by application of Principal Components Analysis. The entire data set is utilized to determine the first two principal components (i.e. PCI and PCII). The scoring coefficients are then utilized to plot PCI vs. PCII for each region. Outliers are determined subjectively. Data points that are determined to be outliers result in invalidation of results.

6.3.1.3 Computerized Data Screening for Cumulative Air Sampling

The first approach applied to data validation is to check the integrity of sample analysis. Sample integrity checks include sample flow volume and sampling period. The second approach is to screen the data for outliers statistically by applying the Dixon Ratio test to the highest and lowest values observed in each region for each sampling

period and those observed in each station. If an outlier is detected a report is generated and the sample is investigated further.

Sample records are qualified by three methods. Field comments are appended to the sample record at the point of collection to reflect the sample quality and possible source of contamination. Office comment codes are appended to sample records to indicate failure of specific integrity checks. If outliers are detected, validation flags may be appended to individual analytical results.

The data validation process incorporates three editing/screening stages. The first stage is an editing process wherein the data output is compared with historical records to ensure accuracy of the dataset. The second stage process utilizes a computerized routine to review each data record and objectively add office comments to indicate failure of specific sample integrity checks (e.g. flow volume and sampling period). The third and final stages of data validation are the subjective review. Statistical procedures such as the Dixon Ratio test are used to select unlikely analytical results. Unreasonable results are either corrected (if typographical) or flagged as unreliable. An analytical result flagged as unreliable may indicate that the reported result appears significantly influenced by contamination (wind blown particules, road salt, etc.) or the reported result appears to reflect

deposition from local sources, contrary to the network objectives. No results are deleted from the database.

## Sample Analysis Integrity Check

### Flow Volume

Flow rate = 2.0 litre/min

Flow volume = 2880 litre/day

The acceptable upper and lower limits for flow volume have been arbitrarily set at 150% and 50% respectively. Flow volume beyond these limits may indicate either instrument malfunction or improper flow rates. Violation of the above limits result in the incorporation of the office comment code "F" into the database.

### Sampling Period

Standard Sampling Period = 28 days

The acceptable upper and lower limits for sampling period are set at 29 and 27 days respectively. Violation of the above limits result in the incorporation of the office comment code "Z" into the database.

Statistical Check for Outliers

The cumulative air monitoring data are grouped by two different methods. In the first method, data are grouped on a regional basis for a given period. In the second method, data are grouped on a station basis. The grouped data are then screened for internal consistency by using the Dixon Ratio test. The Dixon Ratio test (Dixon, 1969, Dean, 1951, Nelson Jr., 1980) is applied to the highest and lowest values with outliers being detected at the 95% level of confidence. This test, however, is limited to data sets with no less than 3 elements. Results determined by the Dixon Ratio test to be outliers are subjectively reviewed prior to flagging the database.

6.3.2 Final Data Screening

The final aspect of the data screening procedures consists of a data check carried out by the regional technicians. At this stage, the preliminary screened data set along with a list of questionable data points is to be returned to the regional technician.

This procedure will prove useful in validation of data and clearing up outstanding problems as well as involving the regional people in the data management QA/QC program. The specific checks that the technicians should perform include:

- clearing up any questions raised by the Database Scientist during data screening;
- confirm any missing data;
- ensuring data are reported for each field sheet submitted;
- looking at unusual data (e.g. poor collection efficiencies, low air sample volumes, etc.).

These technician data checks are to be carried out on a annual basis and should be completed as quickly as possible so that corrective action can be initiated if problem areas are discovered.

6.3.3 Data Evaluation

The data evaluation stage consists of a subjective review of all data by the network quality assurance coordinator. The checks that are to be undertaken at this stage of data validation include:

1) a subjective evaluation of all suspect data points identified in the manual and computerized data screening procedures; this review should result in flagging data points or parameters and invalidating suspect data;
2) a general review of data results by comparing data listings with technician QA summaries, historical data results and station to station comparisons;

3) a confirmation of any missing or unusual field data with original field data sheets;
4) an evaluation of network data in terms of overall data quality;
5) an identification of any corrective action required to correct suspect data or to prevent similar problems from occurring in the future.

It is essential that these subjective data checks by the QA Coordinator take place on a regular basis since these types of checks are an important link in the overall QA/QC systems. Errors detected at this stage will ensure data quality, and avoid the embarrassment of detecting simple, but significant inaccuracies after data have been published.

## 6.4 Data Reporting

Data reports provide the link between the network and data users and must therefore be structured to provide users with the maximum amount of useful information within a reasonable time period.

The database scientist is to establish realistic reporting schedules for each type of data and take steps to ensure that these targets are met.

## 6.5 QA/QC Data Reporting

The QA/QC data reporting requirements have been outlined in a general fashion in Section 3.7. The database and data filing system is to be updated to provide the capability of extracting and processing the QA/QC data on a routine basis. This will require the establishment of a separate QA/QC database containing results of co-located sampling, composite handling, blank analyses, etc. Data manipulation, calculation and plotting routines are to be set up in order to provide the quality assurance coordinator with the means to calculate the data quality parameters described in Sections 4.2.1.4 and 4.2.2.4 on a routine (quarterly) basis. These steps are to be carried out by the QA Coordinator and database scientist.

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# APPENDIX I

## APIOS SITE DESCRIPTION QUESTIONNAIRE

# APIOS SITE DESCRIPTION QUESTIONNAIRE

**Date:** _____

**COMPLETED BY:** _____ (check one)

**REVISION NO:** _____ __ Existing Site (regular network)

__ Potential Site

__ Special Study Site

## 1. SITE IDENTIFICATION

Cumulative Wet _____ Cumulative Dry _____

Event Wet _____ Event Dry _____

Other (describe) _____

**Station Name** _____ **Station Number** _____

**MOE Region** _____ County _____ Township _____

Latitude ____° ____' ____" Longitude ____° ____' ____" Elevation ____m

UTM Co-ordinates _____ E _____ N _____

Name of Primary Operator _____

Alternate Operator _____

Regional Technician _____

Mailing Address (Primary Operator)

_____

_____

_____

Phone (Primary Operator) _____/_____ (Res) _____/_____ (Bus)

Phone (Alternate Operator)_____/_____ (Res) _____/_____ (Bus)

Nearest APIOS Site Cumulative: Station Name _____

Distance _____ km

Event: Station Name _____

Distance _____ km

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## 2. LIST OF INSTRUMENTATION

1) Instrument Type: _____

Cumulative Wet: _____ Cumulative Dry: _____

Event Wet: _____ Event Dry: _____

Other (describe): _____

Manufacturer: _____ Model #: _____

2) Instrument Type: _____

Cumulative Wet: _____ Cumulative Dry: _____

Event Wet: _____ Event Dry: _____

Other (describe): _____

Manufacturer: _____ Model #: _____

3) Instrument Type: _____

Cumulative Wet: _____ Cumulative Dry: _____

Event Wet: _____ Event Dry: _____

Other (describe): _____

Manufacturer: _____ Model #: _____

1) Standard Gauge Type _____

2) Standard Gauge Type _____

Other _____

_____

_____

_____

## 3. SITE LOGISTICS

1) Is road access to site in summer: _____ good _____ fair _____ poor?

winter: _____ good _____ fair _____ poor?

2) Type of road surface? (dirt, gravel, oiled, paved) _____

3) How far is the primary collector from road access? _____ m

4) If necessary, how close can a vehicle approach the collector? _____

5) If no road access to site, how is site reached? (plane, snowmobile) _____

6) What is the available electrical power at site? _____

Volts _____ Amps _____ No. of circuits _____

7) Is the circuit on a Receptacle GFIC _____

Circuit Breaker GFIC _____

No GFIC _____

Battery _____

8) What is the distance from collector receptacle (or proposed site) to circuit panel?

_____m

9) What are the number of power failures per month? _____

per year? _____

10) Are there any other logistical problems which prevent the sampling site being easily approached or operated?

(locked gate, guard dog, flooding in spring?)

_____

## 4. TOPOGRAPHY AND LAND USE

1) Ground slope at site: _____ %

2) Ground cover within 15 m of sampler (grass, scrub, gravel, sand, soil)

_____

3) Soil type within 10 m of site (sand, topsoil, clay, rocky)

_____

Soil type within 500 m of site (sand, topsoil, clay, rocky)

_____

4) Land use % near site, within 1 km/10 km

% Cultivated _____/_____

% Orchard _____/_____

% Lawn _____/_____

% Pasture _____/_____

% Forest _____/_____

% Water _____/_____

% Other (describe) _____/_____

Type of Cultivated crop(s) _____

Forest types within 1 km of site _____

5) What types of windbreaks are within 200 m of primary sampler (buildings, trees, hills) _____

6) Indicate any windbreaks within 200 m of primary sampler (Mark what quadrants of the compass have a windbreak)

7) Prevailing wind direction during event (according to Operator)

Winter _____ °

Summer _____ °

8) Comment on local topography (i.e. flat, hilly, river, basin, etc.)

_____

_____

_____

_____

_____

_____

9) Cultivated Land Use

| Type of Crop | Type of Herbicide or Insecticide Used and when applied | Type of Fertilizers and when applied | Future Crops | Distance to Crops | Direction to Crops |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

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10) Any planned land use in future? _____

_____

_____

11) In winter is there any evidence of drifting snow? _____

12) Tall objects near sampler:

i) Trees _____ (species) Max Height _____ m

Distance _____ m Direction _____ °

ii) Buildings _____ (type) Height _____ m

Use _____ Type of Heating _____ Distance _____ m

Direction _____ °

iii) Other (overhead wires, telephone poles, hedges)

A) Object _____ Height _____ m

Direction _____ ° Distance _____ m

B) Object _____ Height _____ m

Direction _____ ° Distance _____ m

C) Object _____ Height _____ m

Direction _____ ° Distance _____ m

D) Object _____ Height _____ m

Direction _____ ° Distance _____ m

Comments _____

_____

_____

_____

_____

_____

## 5. HUMAN ACTIVITY

1) Large highways (expressways): Distance _____ (Km, m)

Route # _____ Direction from Sampler _____

2) Other paved roads: Distance _____ (Km, m)

Direction from sampler _____

traffic: Heavy _____, Medium _____, Light _____

3) Unpaved road: Distance _____ (Km, m)

Direction from sampler _____

traffic: Heavy _____, Medium _____, Light _____

Surface (dirt, gravel, oiled) _____

4) Parking lot: Distance _____ (Km, m)

Direction from sampler _____

Unpaved _____ Surface material _____ Use: Continuous _____

5) Lake/river or rail traffic: Distance _____ (Km, m)

Direction from sampler _____ barge _____

lake steamer _____ rail _____

traffic: Heavy _____, Medium _____, Light _____

6) Snow clearing procedures:

| Type of surface (road, parking log, hwy, walkway) | Surface distance from sampler | Direction from sampler | Snow Control (plowed, sanded, blown, salted-with what?) |
|---|---|---|---|

7) Airport(s): Distance _____ (Km, m)

Direction from sampler _____ Name _____

traffic: Heavy _____, Medium _____, Light _____

traffic type: _____

8) Stationary sources:

Power plant(s): Distance _____ (Km, m)

Direction from sampler _____ Fuel (gas, coal) _____

Electrical Capacity _____ ($KW_e$, $MW_e$)

Light industry: Name _____ Distance _____ (km)

Direction from sampler _____ Product _____

Heavy industry: Name _____ Distance _____ (km)

Direction from sampler _____ Product _____

9) Other sources: Significant agricultural operations:

Distance _____ (Km, m)

Direction from sampler _____

Other (please describe) _____

_____

Distance _____ (Km, m)

Direction from sampler _____

Other could include - gravel pits, sewage lagoons, marshes, MTC,

Salt or Sand piles - (see Topographical Maps)

10) Effects from local cities, towns, or villages (population)

A ≤ 100,000
B ≤ 10,000
C ≤ 5,000
D ≤ 1,000

| NAME | POPULATION (letter code) | DISTANCE | DIRECTION | TYPES OF POLLUTION SOURCES |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

6. **SITE SUMMARY**

List deviations from site criteria and all advantages and disadvantages of this site.

(use reverse side if necessary)

## 7. SITE LOCATION

1) A topographic map (Scale 1:50,000) indicating sample site with a Red X.

   - arrows on top map indicating best approach from nearest "MAIN" Highway.

   - indicate with red dots any major sources of pollution of contamination (industry, gravel pits, towns)

2) A concise description on how to reach this site. (below)

## 8. SITE DIAGRAM

1) Notable Items (mark all distances)

1. North
2. Direction of prevailing winds
3. Windbreaks
4. Roads (with names)
5. Parking lots
6. Buildings (indicate type of heating)
7. Potential contamination sources
8. Ground cover (grass, scrub, soil, sand)
9. Trees/hedges/marshes
10. Obstructions (include height): poles, towers
11. Topography
12. Water
13. Crops/gardens
14. Paths (foot, snowmobile, ski)
15. Location of existing hydro facilities
16. Position of sampler(s)
17. Nature of soil (rocky, sandy, clay)

2) Legend

Coniferous trees (include height, H= )

Deciduous trees (include height, H= )

Wooden fencing (include height, H= )

Wire fencing (include height, H= )

Grass

Direction of ground slope

Bushes/hedges (include height, H= )

Buildings (height and heating)

Railway tracks

9. SITE PICTURES (Indicates site name, compass point and date of photograph on back of all slides)

1) Two pictures are to be taken so as to best include all sampling instrumentation on site. If possible take pictures 90° apart from each other. Indicate on Site Diagram as P1 and P2 where the pictures were taken from. Also, identify each picture on a list of photographs.

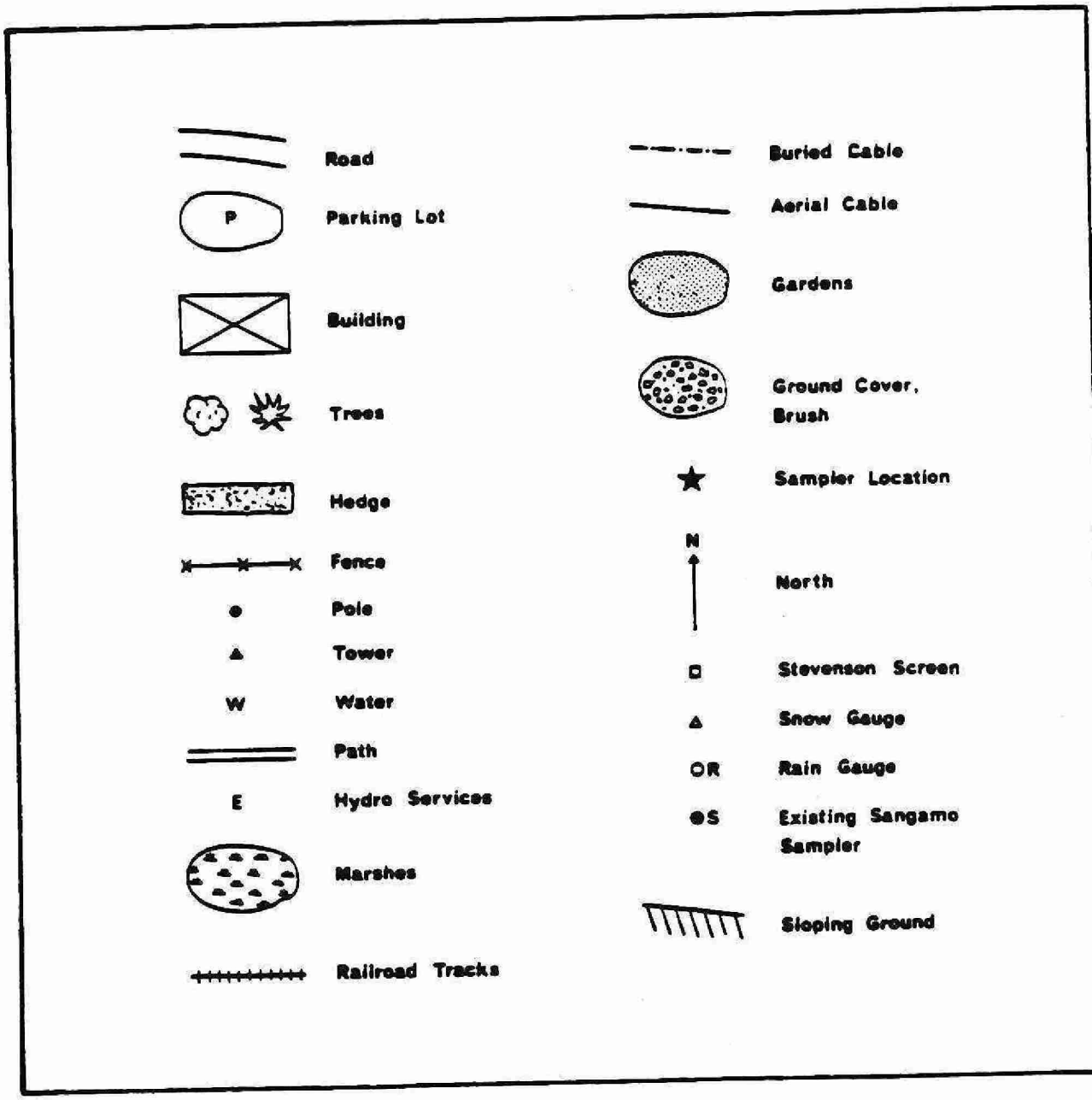
2) Take four pictures showing the area surrounding the site. These should be taken at four compass points from just behind primary sampler facing in the direction of the compass point.

3) Special pictures showing nearby potential sources of contamination (e.g. salt pile) that may affect the site are to be taken. These should be indicated, if possible, on a site diagram as SP1, SP2, etc. with a description of the picture on the back of the print.

LIST OF PHOTOGRAPHS

| # | Direction Facing | Comments |
|---|---|---|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

Legend of symbols for use in site drawings.



# APPENDIX II

## QA/QC RESPONSIBILITIES BY NETWORK POSITION

NETWORK MANAGEMENT

- Have responsibility for ensuring program resources (manpower and financial) are adequate for implementation and execution of network QA/QC prorams.
- Have responsibility for conducting periodic (annual) reviews of network QA/QC program results.
- Have responsibility for initiating and planning of special studies and program changes to increase the level of data recovery and of data quality and the effectiveness of the network QA/QC programs.
- Have responsibility for reviewing the effectiveness of the network QA/QC program with respect to the QA/QC objectives.

QUALITY ASSURANCE COORDINATOR'S RESPONSIBILITIES

- Coordinate regional/head office interactions and set agenda for annual network review.
- Monitor network sample custody procedures - in particular evaluate technician field summary reports for event/cumulative wet/dry networks on a continuous basis, and initiate any corrective action required.
- Design and implement specific training requirements (for new and existing employees) for all areas of the program including:
  - field operations (operator and technician),
  - instrument calibration and repair,
  - data collection, validation and reporting.
- Evaluate the effectiveness of network training procedures and implement corrective action as required.
- Maintain document control system.
- Preparation and distribution of APIOS newsletter for site operators on an quarterly basis.
- Monitor corrective action requirements, maintain the corrective action log and follow up outstanding corrective action requirements.
- Write RFP's for external audits, approve contractor audit plans and monitor audit programs when required.
- Approve mandatory quality assurance plans for network special studies.

- Preparation of QA reports for:

  (1) data users.

  (2) network management.

- Assess impact of site change recorded on site change forms.
- Ensure that procurement, acceptance and instrument pre-testing procedures are properly documented and carried out by the head office environmental technician.
- Monitor network calibration using data provided on calibration report forms.
- Ensure all service and maintenance checks are carried out on time.
- Design and monitor internal auditing and performance evaluation procedures to monitor QA/QC procedures on a regular basis.
- Monitor calibration of ancillary field measurements (volume, field pH, etc.) on a routine basis.
- Use routine QA/QC data to calculate and interpret estimates of data precision accuracy, comparability.
- Subjective review of all data - particularly unusual data points from data screening procedures.

ENVIRONMENTAL TECHNICIAN - TORONTO

- Check in all samples at laboratory reception and perform preliminary QC checks (eg. sample ID, labelling, etc.).
- Carry out instrument procurement, assessment and pre-testing procedures for all instrumentation, equipment and supplies.
- Maintain current copy of Technician Instrument Binder, modified to include procurement and pre-testing procedures.
- Maintain overall network supplies inventory.

REGIONAL TECHNICIAN

- Ensure all information is properly transferred from operator to field submission sheet.
- Prepare monthly field summaries for the event and cumulative sampling networks.
- Train network operators in instrument and sample handling procedures and perform routine evaluations.
- Communicate with operators on a regular basis. Monthly telephone calls (corresponding to changeover days) are recommended.
- Ensure that the field downtime limits of 5 days for event and 10 days for cumulative sites are not exceeded.
- Ensure operators use current operating procedures.
- Initiate corrective action procedures with respect to field operations.
- Carry out annual site evaluations and photograph sites for site review.
- Report any site changes using site change monitoring form.
- Calibrate and maintain network instrumentation.
- Report calibration to network QAC using calibration report forms.
- Maintain regional inventory supply and ensure all sites have 2-3 month supply.
- Ensure operators have properly documented all samples.
- Submit QA/QC samples for processing on a regularly scheduled basis.
- Perform audit checks for in field measurements of sample volume, field pH, etc. as required.

OPERATOR

- Document all relevant sample information on data sheets and log books.
- Be aware of site changes that may affect sample quality and notify technicians whenever they occur.
- Notify technician immediately in case of instrument failure, site problems, etc.
- Avoid potential sample contamination by carefully following sample handling procedures.

LABORATORY QA/QC SCIENTIST

- Checks the suitability of the analytical methods for chemical parameters and recommends changes where necessary.
- Checks the laboratory performance for all the analytical parameters through the mechanism outlined in the QA/QC program.
- Involves the laboratory in interlaboratory comparisons, reports on the results and outlines problem areas for immediate attention.
- Reviews the QA/QC laboratory data summaries on a regular basis for all of the relevant analytical methods, notes significant changes in performance and recommends action on any deterioration in the performance of specific methods.
- Provides QA/QC summaries of analytical method performance on a regular basis and notes any significant difference on the previous performance to QA Coordinator.

DATABASE SCIENTIST

- Check data merging between LIS and SIS data listings.
- Create a QA/QC database by extracting QA/QC data from regular files.
- Develop data manipulation routines to perform calculations, plots, etc. for QA/QC data reporting requirements.
- Train and evaluate data personnel to carry out routine data handling procedures.

# APPENDIX III

## ANALYTICAL METHOD QA SUMMARIES

APPENDIX

TEST NAME:
LAB/UNIT:
LIST TEST NAME CODE:
WORK STATION CODE:
METHOD CODE:

DATE:
IMPLEMENTATION DATE:
UNITS:
UNIT CODE:
SUPERVISOR:

SAMPLE TYPE/MATRIX:

SAMPLE PREPARATION:

SAMPLE HANDLING:
- Quantity Required:
- Container:
- Preservative:
- Other:

ANALYTICAL PROCEDURE:

INSTRUMENTATION:
To include wavelength, flow cell length, temperature, etc.

Operating Scale:
Instrument Detection Limit:

OPERATING CHARACTERISTICS:
Concentration Range: T: W:
F.S.:
Method Detection Limit:
Std. Dev.:
From performance duplicate data @ 3 different concentration ranges
Reporting Increments:

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Reference for peak heights |
| Response Test: | Yes/No |
| Blank: | Chemical used in sample preparation to set proper zero reference (digested) |
| Calibration: | Sequence of low and high calibration standards |
| Calibration Control: | QCA, QCB |
| Recovery Check: | Analysis of reference material or complex std. given as % of expected concentration |
| Drift Controls: | Also called sensitivity - run L and H cal. stds. ~ every 10 samples |
| Duplicates: | |
| Spike Recovery: | Also called 'standard addition' - one of a pair of duplicates is spiked - difference expressed as % |
| Confirmation: | External reference materials, interlab comparisons, another methodology |

All spaces to be filled in. Use NA (not applicable) or UA (unavailable).

TEST NAME: pH

LAB/UNIT: PR

LIS TEST NAME CODE: PH

WORK STATION CODE: PHACD

METHOD CODE: 002AI1

DATE: 821223

IMPLEMENTATION DATE: 770401

UNITS: pH units

UNIT CODE: 000000

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
NA

SAMPLE HANDLING:
Quantity Required: 10 mL
Container: 25x80 mm glass tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
pH is directly measured on a stirred sample (10 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion and temperature range are uniform for all samples and standards.

INSTRUMENTATION:
Radiometer PHM84 pH meter; Ingold #6015-03 electrode with high sensitivity glass for low conductivity solutions; Pet computer system; in-house interface box; in-house software.

Operating Scale: 0-14
Instrument Detection Limit: NA

OPERATING CHARACTERISTICS:
Concentration Range: T: NA W: NA
F.S.: 14.00
Method Detection Limit: NA
Std. Dev.: 0.040 @ 3.000-4.000; 0.0261 @ 4.000-5.000; 0.0539 @ 5.000-6.000
0.0811 @ 6.000-9.000; 0.0492 Overall
Reporting Increments: 0.01

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | NA |
| Response Test: | NA |
| Blank: | Long term DDW bubbled 15 min with N2 |
| Calibration: | Buffer control pH 7.00, slope control pH 4.00 |
| Calibration Control: | QCA buffer pH 6.86, QCB pH 4.008 bubbled with N2 15 |
| Recovery Check: | NA |
| Drift Controls: | NA |
| Duplicates: | Max 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Acidity-Total Fixed Endpoint (TFE) DATE: 840809

LAB/UNIT: PR IMPLEMENTATION DATE: 770401

LIS TEST NAME CODE: ACDT UNITS: mg/L as CaCO3

WORK STATION CODE: PHACD UNIT CODE: 064915

METHOD CODE: 001BT2 SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Domestic Water, Industrial Waste, Sewage, River, Lake, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 10 mL
Container: 25x80 mm glass tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sample aliquots (10 mL) are titrated in an automated titration system with 0.01N sodium hydroxide to a pH endpoint of 8.3. The titrant is standardized against titrating 0.005N KHP of the pH 8.3 endpoint. Titrant delivery is determined from the titration curve derivative and the stability of the pH readings following each aliquot of titrant.

INSTRUMENTATION:
Radiometer PHM84 pH meter; Ingold #6015-03 electrode with high sensitivity glass for low conductivity solutions; ABU80 autoburette; Pet computer system; in-house interface box; in-house software.

Operating Scale: NA
Instrument Detection Limit: 0.01 mg/L

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.20 mg/L W: 0.01 mg/L
F.S.: 50.0 mg/L
Method Detection Limit: 0.40 mg/L
Std. Dev.: 0.1221 @ <2.000; 0.2241 @ 2.000-5.000; 0.1750 @ 5.000-10.000; 0.5763 @ 10.000-25.000; 0.9886 @ 25.000-100.0
Reporting Increments: 0.01 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | NA |
| Response Test: | NA |
| Blank: | Long term DDW bubbled 15 min with N2 |
| Calibration: | 0.005N KHP bubbled 15 min N2; buffer 6.86; buffer 4.008 |
| Calibration Control: | QCA 0.0005N KHP; QCB 0.0002N H2SO4 bubbled 15 min N2 |
| Recovery Check: | NA |
| Drift Controls: | NA |
| Duplicates: | Max 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Acidity-Gran Analysis

DATE: 840809

LAB/UNIT: PR

IMPLEMENTATION DATE: 820615

LIS TEST NAME CODE: ACDG

UNITS: mg/L as H+

WORK STATION CODE: PHACD

UNIT CODE: 063801

METHOD CODE: 001BT5

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 10 mL
Container: 25x80 mm glass tubes
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sample aliquots (10 mL) are titrated in an automated titration system with 0.01N sodium hydroxide to a pH endpoint of>8.3. The titrant is standardized against titrating 0.005N KHP of the pH 8.3 endpoint. Titrant delivery is determined from the titration curve first derivative and the stability of the pH readings following each aliquot of titrant. Data is subject to Gran analysis to determine the total acidity component.

INSTRUMENTATION:
Radiometer PHM84 pH meter; Ingold #6015-03 electrode with high sensitivity glass for low conductivity solutions; Pet computer system; in-house interface box; in-house software.

Operating Scale: NA
Instrument Detection Limit: 0.01

OPERATING CHARACTERISTICS:
Concentration Range: T: 2.0 ug/L W: 0.1 ug/L
F.S.: 1000ug/L
Method Detection Limit: 4.0 ug/L
Std. Dev.: UA

Reporting Increments: 0.1 ug/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | NA |
| Response Test: | NA |
| Blank: | Long term DDW bubbled 15 min with N2 |
| Calibration: | 0.005N KHP; buffer 6.86; buffer 4.008 |
| Calibration Control: | QCA 0.0005N KHP; QCB 0.0002N H2SO4 |
| Recovery Check: | NA |
| Drift Controls: | NA |
| Duplicates: | Max 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Nitrogen-Nitrate

DATE: 821213

LAB/UNIT: PR

IMPLEMENTATION DATE: 770401

LIS TEST NAME CODE: NNO3UR

UNITS: mg/L as N

WORK STATION CODE: PRIC1

UNIT CODE: 064807

METHOD CODE: 003AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
Automated addition of $Na_2CO_3/NaHCO_3$ to match the eluent strength, due to simultaneous chloride analysis.

SAMPLE HANDLING:
Quantity Required: 10-15 mL
Container: 16x100mm polystyrene tubes
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of nitrate is determined by comparison of sample scan to a series of standard scans. Chloride and sulphate are measured simultaneously.

INSTRUMENTATION:
Dionex Ion Chromatograph Model 10 modified to be compatible with automated sampling train; Technicon sampler; AA1 peristaltic pump; timer (Supergrator III); in-house control box; Linear recorder.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.05 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.02 mg/L W: 0.01 mg/L
F.S.: 2.00 mg/L
Method Detection Limit: 0.04 mg/L
Std. Dev.:

Reporting Increments: 0.01 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity. |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.20, 0.40, 0.80, 1.20, 1.60, 2.00 mg/L standards |
| Calibration Control: | QCA 1.60 mg/L, QCB 0.40 mg/L |
| Recovery Check: | NA |
| Drift Controls: | 0.80 mg/L cal. std. in run |
| Duplicates: | 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sulphate

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: SSO4FR

UNITS: ug/Filter as SO4

WORK STATION CODE: PRSEQ

UNIT CODE: 361941

METHOD CODE: 004AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Teflon filters from sequential filter packs.

SAMPLE PREPARATION:
Filters are extracted with DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 25 mL extractant.
Container: 25x90 mm capped polystyrene tube; 12x100 mm polystyrene tube.
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sulphate is separated from other anions in the sample by automated, suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate is determined by comparison of the sample scan to a series of standard scans. Results are converted to and reported as ug/Filter as SO4.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system A); Gilson sampler; Technicon AAI pump; Linear recorder. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 umho/cm
Instrument Detection Limit: 0.01 umho/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 1.25 ug/Filter
F.S.: 250 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 1.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 mg/L as SO4 stds. |
| Calibration Control: | QCA 8.00, QCB 2.00 mg/L as SO4 |
| Recovery Check: | NA |
| Drift Controls: | 8.00 mg/L std. every 10 samples. |
| Duplicates: | 2 per day |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sulphur Dioxide as SO2

DATE: 840809

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: SSO2FR

UNITS: ug/Filter as SO2

WORK STATION CODE: PRSEQ, PRLOV

UNIT CODE: 361943

METHOD CODE: 004AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Impregnated W41 filters from Lo-Vol and Sequential filter packs.

SAMPLE PREPARATION:
W40 filters are extracted with 0.05% H2O2 with mechanical shaking, ultrasound treatment to enhance extraction, followed by a 24 hour rest period. SO2 is converted to SO4 in the process.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: Filter impregnated with K2CO3 and Glycerol by field staff

ANALYTICAL PROCEDURE:
Sulphate is separated from other anions in the sample by automated, suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate is determined by comparison of the sample scan to a series of standard scans. Results are converted to and reported as ug/Filter as SO2.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system B); Gilson sampler; Technicon AAI pump; Linear recorder. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 umho/cm
Instrument Detection Limit: 0.01 umho/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: 4.65 ug/Filter W: 1.65 ug/Filter
F.S.: 333.5 ug/Filter
Method Detection Limit: 9.3 ug/Filter as SO2
Std. Dev.: UA

Reporting Increments: 1.65 ug/Filter as SO2

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 mg/L as SO4 stds. |
| Calibration Control: | QCA 8.00, QCB 2.00 mg/L as SO4 |
| Recovery Check: | NA |
| Drift Controls: | 4.00 mg/L std. every 10 samples. |
| Duplicates: | One to three split entracts per run. |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sulphate

LAB/UNIT: PR

LIS TEST NAME CODE: SSO4NF

WORK STATION CODE: PRSEQ

METHOD CODE: 004AI0

DATE: 840809

IMPLEMENTATION DATE: 840330

UNITS: ug/Filter as SO4

UNIT CODE: 361941

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Nylon filter from Sequential filter packs.

SAMPLE PREPARATION:
Nylon filters are extracted with 0.003N NaOH with ultrasound treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 25 mL extractant
Container: 25x90 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sulphate is separated from other anions in the sample by automated, suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate is determined by comparison of the sample scan to a series of standard scans. Results are converted to and reported as ug/Filter as SO4.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system A); Gilson sampler; Technicon AAI pump; Linear recorder. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 umho/cm
Instrument Detection Limit: 0.01 umho/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 1.25 ug/Filter
F.S.: 250 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 1.25 ug/Filter as SO2

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 mg/L as SO4 stds. |
| Calibration Control: | QCA 8.00, QCB 2.00 mg/L as SO4 |
| Recovery Check: | NA |
| Drift Controls: | 4.00 mg/L std. every 10 samples. |
| Duplicates: | Minimum one per day. |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sulphate

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: SSO4UR

UNITS: ug/Filter as SO4

WORK STATION CODE: PRLOV

UNIT CODE: 361941

METHOD CODE: 004A10

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
W40 filters from Lo-Vol filter packs.

SAMPLE PREPARATION:
Filters are extracted with DDW with ultrasound treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sulphate is separated from other anions in the sample by automated, suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate is determined by comparison of the sample scan to a series of standard scans. Results are converted to and reported as ug/Filter as SO4.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system A); Gilson sampler; Technicon AAI pump; Linear recorder; Fast-Run Columns. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.01 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 2.5 ug/Filter
F.S.: 500 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 2.5 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 1.00, 2.00, 4.00, 6.00, 8.00, 10.00 mg/L as SO4 stds. |
| Calibration Control: | QCA 8.00, QCB 2.00 mg/L as SO4 |
| Recovery Check: | NA |
| Drift Controls: | 4.00 mg/L std. every 10 samples. |
| Duplicates: | 2 per day |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sulphate

DATE: 840809

LAB/UNIT: PR

IMPLEMENTATION DATE: 770401

LIS TEST NAME CODE: SSO4UR

UNITS: mg/L as SO4

WORK STATION CODE: PRIC1

UNIT CODE: 064941

METHOD CODE: 003AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
Automated addition of $Na_2CO_3/NaHCO_3$ to match the eluent strength, due to simultaneous chloride analysis.

SAMPLE HANDLING:
Quantity Required: 10-15 mL
Container: 16x100 mm polystyrene tubes
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Sulphate is separated from other anions in the sample by automated, suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate is determined by comparison of the sample scan t a series of standard scans. Chloride and nitrate are analyzed simultaneously.

INSTRUMENTATION:
Dionex Ion Chromatograph Model 10 modified to be compatible with an automated sampling train. Technicon sampler, AA1 peristaltic pump; timer (Supergrator III); control box (in-house design); Linear recorder.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.05 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.06 mg/L W: 0.01 mg/L
F.S.: 10.0 mg/L
Method Detection Limit: 0.12 mg/L
Std. Dev.: UA @ <2.00; 0.064 @ 2.00-5.00; 0.116 @ 5.00-10.00

Reporting Increments: 0.01 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 1.0, 2.0, 4.0, 6.0, 8.0, 10.0 mg/L stds. |
| Calibration Control: | QCA 8.0 mg/L, QCB 2.0 mg/L |
| Recovery Check: | NA |
| Drift Controls: | 6.0 mg/L cal. std. in run |
| Duplicates: | 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Conductivity

DATE: 840809

LAB/UNIT: PR

IMPLEMENTATION DATE: 831001

LIS TEST NAME CODE: COND25

UNITS: uS/cm at 25C

WORK STATION CODE: PRIC1

UNIT CODE: 350351

METHOD CODE: 002AI2

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
NA

SAMPLE HANDLING:
Quantity Required: 10-15 mL
Container: 16x100 mm polystyrene tubes
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
A sample stream is diverted from the automated ion chromatograph sampling system and is introduced into a jacketed conductivity cell and equilibrated to 25C. Conductivity is read from an attached chart recorder calibrated against the meter scale.

INSTRUMENTATION:
Radiometer CDM83 Conductivity meter with CDC334 jacketed conductivity cell. Temperature controlled by a Lauda K2 circulating water bath. Linear recorder.

Operating Scale: 0-150 uS/cm
Instrument Detection Limit: 1 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.1 uS/cm
F.S.: Lower 0-50;upper 0-100uS/cm
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.1 uS/cm

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Meter zero |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | Recorder matched to std resistance meter readings. |
| Calibration Control: | QCA 42 uS/cm; QCB 11 uS/cm mixed ion solutions |
| Recovery Check: | NA |
| Drift Controls: | Conductivity of 40% F.S. mixed ion soln, about 23 uS/cm |
| Duplicates: | 2 per day |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

Concord Scientific Corporation

TEST NAME: Nitrogen- NH3 and NH4+

LAB/UNIT: PR

LIS TEST NAME CODE: NNHTFR, NNHTUR

WORK STATION CODE: PRNUT

METHOD CODE: 103CC2

DATE: 840810

IMPLEMENTATION DATE: 840514

UNITS: mg/L as N

UNIT CODE: 064807

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
No samples are filtered.

SAMPLE HANDLING:
Quantity Required: 5 mL
Container: 12x100 mm polyethylene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream which differs from the sample stream by replacement of the catalyst with an equal flow of distilled water is employed to blank out the effects of sample colour.

INSTRUMENTATION:
AAII AutoAnalyzer system, heating bath module (7.7 mL delay coil at 38C); Flow cells=10 mm; 630 nm. 5X expansion at the recorder on low range.

Operating Scale: 0-0.2 Abs low range; 0-1.2 Abs high range
Instrument Detection Limit: 0.01 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.005 mg/L
F.S.: Ranges to 1.00 & 5.00 mg/L
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.005 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.10, 0.20, 0.80, 4.00 mg/L as N stds |
| Calibration Control: | QCA 4.00, QCB 0.80, QCC 0.20 mg/L as N |
| Recovery Check: | NA |
| Drift Controls: | DDW, 0.20, 0.80, 4.00 mg/L stds |
| Duplicates: | At least 3 pairs run randomly in each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Sample colour, eliminated by reference channel |

TEST NAME: Nitrogen- NH3 and NH4+

LAB/UNIT: PR

LIS TEST NAME CODE: NNHTFR

WORK STATION CODE: PRSEQ

METHOD CODE: 004AI0

DATE: 840810

IMPLEMENTATION DATE: 840514

UNITS: ug/Filter as N

UNIT CODE: 361807

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Teflon filters from sequential filter packs.

SAMPLE PREPARATION:
Filters are extracted with DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 25 mL DDW
Container: 25x90 mm capped polystyrene tube; 12x100 mm polystyrene tube.
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream which differs from the sample stream by replacement of the catalyst with an equal flow of distilled water is employed to blank out the effects of sample colour.

INSTRUMENTATION:
AAII AutoAnalyzer system, heating bath module (7.7 mL delay coil at 38C); Flow cells=10 mm; 630 nm. 5X expansion at the recorder on low range.

Operating Scale: 0-0.2 Abs low range; 0-1.2 Abs high range
Instrument Detection Limit: 0.005 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.25 ug/Filt
F.S.: Lower 50 ug/Filt; upper 250
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Fresh DDW |
| Response Test: | NA |
| Blank: | Fresh DDW |
| Calibration: | 0.10, 0.20, 0.80, 4.00 mg/L as N stds |
| Calibration Control: | QCA 4.00, QCB 0.80, QCC 0.20 mg/L as N |
| Recovery Check: | NA |
| Drift Controls: | DDW, 0.20, 0.80, 4.00 mg/L stds |
| Duplicates: | At least 3 pairs run randomly in each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Sample colour, eliminated by reference channel |

TEST NAME: Nitrogen- Nitrate

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: NNO3FR

UNITS: ug/Filter as N

WORK STATION CODE: PRSEQ

UNIT CODE: 361807

METHOD CODE: 004A10

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Teflon filters from sequential filter packs.

SAMPLE PREPARATION:
Filters are extracted with DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 25 mL extractant
Container: 25x90 mm capped polystyrene tube; 12x100 mm polystyrene tube.
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of nitrate is determined by comparison of sample scan to a series of standard scans. Results are converted to and reported as ug/filter N.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system A); Gilson sampler; Technicon AAI pump; Linear recorder; Fast-Run Columns. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 umho/cm
Instrument Detection Limit: 0.01 umho/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.25 ug/Filter
F.S.: 50 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.20, 0.40, 0.80, 1.20, 1.60, 2.00 mg/L N |
| Calibration Control: | QCA 1.60, QCB 0.40 mg/L N |
| Recovery Check: | NA |
| Drift Controls: | 0.80 mg/L N std every 10 samples |
| Duplicates: | 2 per day |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Nitrogen- Nitrate

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: NNO3UR

UNITS: ug/Filter as N

WORK STATION CODE: PRLOU

UNIT CODE: 361807

METHOD CODE: 004AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
W40 filters from Lo-Vol filter packs.

SAMPLE PREPARATION:
Filters are extracted with DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm capped polystyrene tube; 12x100 mm polystyrene tube.
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of nitrate is determined by comparison of sample scan to a series of standard scans. Results are converted to and reported as ug/filter N.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system A); Gilson sampler; Technicon AAI pump; Linear recorder; Fast-Run Columns. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 umho/cm
Instrument Detection Limit: 0.01 umho/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.50 ug/Filter
F.S.: 100 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.20, 0.40, 0.80, 1.20, 1.60, 2.00 mg/L N |
| Calibration Control: | QCA 1.60, QCB 0.40 mg/L N |
| Recovery Check: | NA |
| Drift Controls: | 0.80 mg/L N std every 10 samples |
| Duplicates: | 2 per day |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Chloride

DATE: 821224

LAB/UNIT: PR

IMPLEMENTATION DATE: 770401

LIS TEST NAME CODE: CLIDUR

UNITS: mg/L as Cl-

WORK STATION CODE: PRIC1

UNIT CODE: 064817

METHOD CODE: 003AI0

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow, Fish Tissue, Sera

SAMPLE PREPARATION:
Automated addition of Na2CO3/NaHCO3 to match the eluent strength.

SAMPLE HANDLING:
Quantity Required: 10-15 mL
Container: 16x100mm polystyrene tubes
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of chloride is determined by comparison of sample scan to a series of standard scans. Nitrate and sulphate are measured simultaneously.

INSTRUMENTATION:
Dionex Ion Chromatograph Model 10 modified to be compatible with automated sampling train; Technicon sampler; AA1 peristaltic pump; timer (Supergrator III); in-house control box; Linear recorder.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.05 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.03 mg/L W: 0.01 mg/L
F.S.: 1.50 mg/L
Method Detection Limit: 0.06 mg/L
Std. Dev.: 0.0198 @ <0.300; 0.0211 @ 0.300-0.750; 0.0191 @ 0.750-1.500

Reporting Increments: 0.01 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity. |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.15, 0.30, 0.60, 0.90, 1.20, 1.50 mg/L standards |
| Calibration Control: | QCA 1.20 mg/L, QCB 0.30 mg/L |
| Recovery Check: | NA |
| Drift Controls: | 0.60 mg/L cal. std. in run |
| Duplicates: | 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Water dip eliminated by sample preparation. |

TEST NAME: Chloride

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 840330

LIS TEST NAME CODE: CLIDUR

UNITS: ug/Filter as Cl-

WORK STATION CODE: PRLOV

UNIT CODE: 361817

METHOD CODE: 004AIC

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
W40 filter from Lo-Vol filter pack

SAMPLE PREPARATION:
W40 filter is extracted in DDW with ultrasonic treatment followed by a 24 hour rest period. Automated addition of Na2CO3/NaHCO3 "spike" to match eluent strength.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of chloride is determined by comparison of sample scan to a series of standard scans. Results are converted to and reported as ug/filter as Cl-.

INSTRUMENTATION:
Automated Dionex Ion Chromatograph Model 16 (system A); Gilson sampler, Technicon AA1 pump; Linear recorder. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.01uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.50 ug/Filt
F.S.: 75.0 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.50 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity. |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.15, 0.30, 0.60, 0.90, 1.20, 1.50 mg/L standards |
| Calibration Control: | QCA 1.20 mg/L, QCB 0.30 mg/L |
| Recovery Check: | NA |
| Drift Controls: | 0.60 mg/L cal. std.every 10 samples |
| Duplicates: | 2 daily |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Water dip eliminated by automated "spike" addition. |

TEST NAME: Nitrogen-Nitric Acid

LAB/UNIT: PR

LIS TEST NAME CODE: NNRICF

WORK STATION CODE: PRLOV, PRSEQ

METHOD CODE: 004AI0

DATE: 840810

IMPLEMENTATION DATE: 840330

UNITS: ug/Filter as N

UNIT CODE: 361807

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Nylon filters from Lo-Vol and Sequential filter packs.

SAMPLE PREPARATION:
Nylon filters are extracted with 0.003N NaOH with ultrasound treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 25 mL extractant
Container: 25x90 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of nitrate is determined by comparison of sample scan to a series of standard scans. Results are converted to and reported as ug/filter N.

INSTRUMENTATION:
Automated Dionex Ion Chromatography Model 16 (system B); Gilson sampler; Technicon AAI pump; Linear recorder; Fast-Run Columns. System timing taken from Supergrator III and Gilson sampler.

Operating Scale: 0-10 uS/cm
Instrument Detection Limit: 0.01 uS/cm

OPERATING CHARACTERISTICS:
Concentration Range: T: UA W: 0.25 ug/Filter
F.S.: 50 ug/Filter
Method Detection Limit: UA
Std. Dev.: UA

Reporting Increments: 0.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | Suppressed eluent conductivity |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.20, 0.40, 0.80, 1.20, 1.60, 2.00 mg/L NO3-N |
| Calibration Control: | QCA 1.60, QCB 0.40 mg/L NO3-N |
| Recovery Check: | NA |
| Drift Controls: | 0.80 mg/L NO3-N std every 10 samples |
| Duplicates: | Minimum one per run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | NA |

TEST NAME: Sodium

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 790401

LIS TEST NAME CODE: NAUR

UNITS: mg/L as Na

WORK STATION CODE: PRAA

UNIT CODE: 064811

METHOD CODE: 002EA1

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 5 mL
Container: 25x90 mm polystyrene disposable tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Potassium, a suppressing agent, is introduced by an automated sampling train. Chart recorder results are converted to concentration using manually prepared calibration curves.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.5 Abs
Instrument Detection Limit: 0.01 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.019 mg/L W: 0.005mg/L
F.S.: 1.00 mg/L
Method Detection Limit: 0.038 mg/L
Std. Dev.: 0.0115 @ <0.,200; 0.0062 @ 0.200-0.500; 0.0133 @ 0.500-1.000;
0.0110 Overall
Reporting Increments: 0.005 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + reagent (KCl) |
| Response Test: | NA |
| Blank: | DDW + reagent (KCl) |
| Calibration: | 0.10, 0.20, 0.40, 0.60, 0.80, 1.00 mg/L |
| Calibration Control: | QCA 0.60, QCB 0.10 mg/L run after stds. |
| Recovery Check: | NA |
| Drift Controls: | Run B1, 0.20, 0.80 mg/L stds every 10 samples |
| Duplicates: | Run 3 pairs at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Ionization, eliminated by addition of KCl suppressant |

TEST NAME: Sodium

LAB/UNIT: PR

LIS TEST NAME CODE: NAUR

WORK STATION CODE: PRLOV

METHOD CODE: 004AA3

DATE: 840810

IMPLEMENTATION DATE: 820401

UNITS: ug/Filter as Na

UNIT CODE: 361811

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
W40 filter from Lo-Vol filter pack

SAMPLE PREPARATION:
W40 filter is extracted in DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 589.0 nm with an air-acetylene flame. Potassium, a suppressing agent, is introduced by an automated sampling train. Chart recorder results are converted to concentration using manually prepared calibration curves.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.5 Abs
Instrument Detection Limit: 0.001 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.95 ug/Filt W: 0.25 ug/Filt
F.S.: 50 ug/Filter
Method Detection Limit: 1.90 ug/Filter
Std. Dev.: 0.55 ug/Filter Overall

Reporting Increments:

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + reagent (KCl) |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.10, 0.20, 0.40, 0.60, 0.80, 1.00 mg/L |
| Calibration Control: | QCA 0.60, QCB 0.10 mg/L run after stds. |
| Recovery Check: | NA |
| Drift Controls: | Run B1, 0.20, 0.80 mg/L stds every 10 samples |
| Duplicates: | Run 3 pairs at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Ionization, eliminated by addition of KCl suppressant |

TEST NAME: Potassium

LAB/UNIT: PR

LIS TEST NAME CODE: KKUR

WORK STATION CODE: PRAA

METHOD CODE: 002EA1

DATE: 840810

IMPLEMENTATION DATE: 790401

UNITS: mg/L as K

UNIT CODE: 064819

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 5 mL
Container: 25x90 mm polystyrene disposable tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium, a suppressing agent, is introduced by an automated sampling train. Chart recorder results are converted to concentration using manually prepared calibration curves.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.4 Abs
Instrument Detection Limit: 0.001 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.014 mg/L W: 0.005 mg/L
F.S.: 1.00 mg/L
Method Detection Limit: 0.028 mg/L
Std. Dev.: 0.0085 @ <0.200; 0.0137 @ 0.200-0.500; 0.0439 @ 0.500-1.000; 0.0143 Overall
Reporting Increments: 0.005 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + reagent (CsCl) |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.10, 0.20, 0.40, 0.60, 0.80, 1.00 mg/L |
| Calibration Control: | QCA 0.60, QCB 0.10 mg/L |
| Recovery Check: | NA |
| Drift Controls: | Run B1, 0.20, 0.80 mg/L stds every 10 samples |
| Duplicates: | Run 3 pairs at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Ionization, eliminated by addition of CsCl suppressant |

TEST NAME: Potassium

LAB/UNIT: PR

LIS TEST NAME CODE: KKUR

WORK STATION CODE: PRLOV

METHOD CODE: 004BA3

DATE: 840810

IMPLEMENTATION DATE: 820401

UNITS: ug/Filter as K

UNIT CODE: 361819

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
W40 filter from Lo-Vol filter pack

SAMPLE PREPARATION:
W40 filter is extracted in DDW with ultrasonic treatment followed by a 24 hour rest period.

SAMPLE HANDLING:
Quantity Required: 1 filter; 50 mL extractant
Container: 30x120 mm polyethylene capped tube; 12x100 mm polystyrene tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 766.5 nm with an air-acetylene flame. Cesium, a suppressing agent, is introduced by an automated sampling train. Chart recorder results are converted to concentration using manually prepared calibration curves. Results are converted to and reported as ug/Filter as K.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.4 Abs
Instrument Detection Limit: 0.001 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.70 ug/Filt W: 0.25 ug/Filt
F.S.: 50 ug/Filter
Method Detection Limit: 1.40 ug/Filter
Std. Dev.: 0.715 ug/Filter Overall

Reporting Increments: 0.25 ug/Filter

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + reagent (CsCl) |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.10, 0.20, 0.40, 0.60, 0.80, 1.00 mg/L |
| Calibration Control: | QCA 0.60, QCB 0.10 mg/L |
| Recovery Check: | NA |
| Drift Controls: | Run B1, 0.20, 0.80 mg/L stds every 10 samples |
| Duplicates: | Run 3 pairs at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Ionization, eliminated by addition of CsCl suppressant |

TEST NAME: Magnesium

DATE: 840810

LAB/UNIT: PR

IMPLEMENTATION DATE: 790401

LIS TEST NAME CODE: MGUR

UNITS: mg/L as Mg

WORK STATION CODE: PRAA

UNIT CODE: 064812

METHOD CODE: 001CA1

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 5 mL
Container: 25x90 mm polystyrene disposable tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 285.2 nm with an air-acetylene flame. Lanthanum, a releasing agent, in dilute HCl solution is added by an automated sampling train. Chart recorder results are converted to concentration by means of manually prepared calibration curves.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.4 Abs
Instrument Detection Limit: 0.001 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.007 mg/L W: 0.005 mg/L
F.S.: 0.500 mg/L
Method Detection Limit: 0.014 mg/L
Std. Dev.: 0.0043 @ <0.100; 0.0133 @ 0.100-0.250; 0.0078 @ 0.250-0.500

Reporting Increments:

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + Reagent (La2Cl) |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.050, 0.100, 0.200, 0.300, 0.400, 0.500 mg/L standards |
| Calibration Control: | QCA 0.300 mg/L, QCB 0.050 mg/L run after cal. stds. |
| Recovery Check: | NA |
| Drift Controls: | Baseline, 0.100, 0.400 mg/L cal. stds. every 10 samples |
| Duplicates: | 3 pairs run at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Eliminated by addition of releasing agent (La2Cl) |

TEST NAME: Calcium

LAB/UNIT: PR

LIS TEST NAME CODE: CAUR

WORK STATION CODE: PRAA

METHOD CODE: 002CA1

DATE: 840810

IMPLEMENTATION DATE: 790401

UNITS: mg/L as Ca

UNIT CODE: 064820

SUPERVISOR: M. Rawlings

SAMPLE TYPE/MATRIX:
Precipitation, Throughfall, Stemflow

SAMPLE PREPARATION:
None

SAMPLE HANDLING:
Quantity Required: 5 mL
Container: 25x90 mm polystyrene disposable tube
Preservative: NA
Other: NA

ANALYTICAL PROCEDURE:
Samples are analyzed by AAS at 422.7 nm with an air-acetylene flame. Lanthanum (in dilute HCl solution), a releasing agent, is introduced with an automated sampling train. Chart recorder results are converted by means of manually prepared calibration curves.

INSTRUMENTATION:
Technicon sampler; Gilson perstaltic pump; Varian AA-275(AAS), Linear recorder.

Operating Scale: 0-0.17 Abs
Instrument Detection Limit: 0.001 Abs

OPERATING CHARACTERISTICS:
Concentration Range: T: 0.04 mg/L W: 0.01 mg/L
F.S.: 2.00 mg/L
Method Detection Limit: 0.08 mg/L
Std. Dev.: 0.023 @ <0.40; 0.035 @ 0.040-1.00; 0.029 @ 1.00-2.00

Reporting Increments: 0.01 mg/L

CONTROL SEQUENCES:

| | |
|---|---|
| Baseline: | DDW + Reagent (La2Cl) |
| Response Test: | NA |
| Blank: | DDW |
| Calibration: | 0.20, 0.40, 0.80, 1.20, 1.60, 2.00 mg/L standards |
| Calibration Control: | QCA 1.20 mg/L, QCB 0.20 mg/L run after cal. stds. |
| Recovery Check: | NA |
| Drift Controls: | 0.40 and 1.60 mg/L cal. stds. run every 10 samples |
| Duplicates: | 3 pairs run at beginning or end of each run |
| Spike Recovery: | NA |
| Confirmation: | NA |
| Interferences: | Eliminated by addition of releasing agent (La2Cl) |

# APPENDIX IV

# AUDIT QUESTIONNAIRES

CUMULATIVE PRECIPITATION AUDIT - SANGAMO/MIC

OPERATOR: _____

SITE: _____

| Operator's Duties | Yes | No |
|---|---|---|
| 1. What time does operator change sample bags? If he can't change at the proper time, what does he do? (phone technican, change bag when he can, contact alternate operator). _____ _____ _____ | | |
| 2. Does the operator, while removing the sample bag from the sample bucket, allow for any chance of contamination of the precipitation sample? Several points that may present contamination are: | | |
| - is sample bucket removed from sampler prior to changing sample bag? | | |
| - does the operator wear clean, new gloves to remove the knife edge? | ___ | ___ |
| - does he handle only the outside of bucket and knife edge? | ___ | ___ |
| - is he careful not to contaminate knife edge when its taken off? | ___ | ___ |
| - is bag removed in such a way as to not introduce contamination from top and outside portions of bag? | ___ | ___ |
| - is operator careful not to stand directly over pail? | ___ | ___ |
| - is there a potential for contamination by hair, coat, etc.? | ___ | ___ |
| Comments: _____ _____ _____ _____ | | |

| Operator's Duties | Yes | No |
|---|---|---|
| 3. Are the sample bags properly handled to prevent damage, sample loss and allow for proper handling by laboratory staff? Several points should be checked: | | |
| - is air forced out of bag before sealing? | ___ | ___ |
| - is bag tied near enough to the top so that it can be leached for metals analysis, but far enough down so that none of the contaminated surfaces are exposed to sample? How is bag tied and sealed? _____ _____ | ___ | ___ |
| - does operator use a container to store and transport sample? _____ | ___ | ___ |
| - are bags promptly labelled (What information is recorded on label)? _____ _____ | ___ | ___ |
| - for winter operation with long tube how is bag removed? _____ _____ | | |
| - where does operator store sample for pick up by technician? (winter and summer). _____ _____ | | |
| - how long does it take the technician to come and pick up samples? _____ _____ | | |
| Comments: _____ _____ _____ _____ | | |

CUMULATIVE PRECIPITATION AUDIT - SANGAMO/MIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| 4. Are the sampler components carefully cleaned? Several points to be checked: | | |
| - does operator clean the following before putting in new bag: | | |
| Sensor grids with $H_2O$ and ETOH | ___ | ___ |
| Hood gasket with $H_2O$ and ETOH | ___ | ___ |
| - does he use new gloves and Kimwipes for above? _____ | | |
| - what is the cleaning procedure under cold conditions? _____ _____ | | |
| Comments: _____ _____ _____ _____ | | |
| 5. What problems does the operator experience with the sampler? How does he handle them? Some things that can happen: | | |
| - does wet sample bag get stuck in container when being removed? _____ _____ | ___ | ___ |
| - what does operator do if this happens? _____ _____ _____ | | |
| - what procedure is followed if the bag comes out damaged? _____ _____ _____ | | |

| Operator's Duties | Yes | No |
|---|---|---|
| - how often are leaks observed in bags? _____ | | |
| _____ | | |
| - what does he think causes it? _____ | | |
| _____ | | |
| Comments: _____ | | |
| _____ | | |
| _____ | | |
| _____ | | |
| 6. In placing a new sample bag in the collector, does the operator observe the following points: | | |
| - are new gloves worn when touching inside of bag? | ___ | ___ |
| - is operator careful not to touch inside of bag any more than is necessary? | ___ | ___ |
| - is bag forced to bottom of container by filling with air? _____ | ___ | ___ |
| _____ | | |
| - is operator careful not to put hand inside bag when installing it? | ___ | ___ |
| - is enough bag left over the edge to permit knife edge to hold bag? | ___ | ___ |
| - are new gloves used to flatten bag against the pail? | ___ | ___ |
| - is collar cleaned, rinsed and dried before replacing on container? | ___ | ___ |

| Operator's Duties | Yes | No |
|---|---|---|
| - Does the end result produce a well fitting bag? (smooth surface, no overhang). _____ | | |
| Comments: _____ | | |
| 7. Does the operator have a good knowledge of what he is expected to do? Specific points that the operator should be asked: | | |
| - does operator have written instructions for procedures? | ___ | ___ |
| - which procedures does he have? _____ | | |
| - does he think he was adequately trained? _____ | ___ | ___ |
| - when was the last time the regional technician reviewed procedures with him? _____ | | |
| - does he have any particular problems with the sampling? _____ | ___ | ___ |

CUMULATIVE PRECIPITATION AUDIT - SANGAMO/MIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| Comments: _____ | | |
| 8. Does the operator have sufficient supplies on site? He should be asked: | | |
| - does he receive regular shipment of supplies or as needed? _____ | | |
| - does he have enough of the following: | | |
| Sample forms | ___ | ___ |
| Bags | ___ | ___ |
| DI $H_2O$ and ethanol | ___ | ___ |
| Kimwipes | ___ | ___ |
| Gloves | ___ | ___ |
| - has he ever run out of supplies? | ___ | ___ |
| Comments: _____ | | |
| 9. What comments does the operator have about the programme? | | |
| - does the operator understand why he is collecting the samples? _____ | ___ | ___ |

| Operator's Duties | Yes | No |
|---|---|---|
| - does operator understand why all the special precautions (glove, cleaning bags etc.) are required? _____ | | |
| - does operator get any feedback on programme? | ___ | ___ |
| - Would he like feedback? How often? _____ | ___ | ___ |
| - does he have any special comments with regards to the sampling, site, technician or programme as a whole? _____ | | |
| - Does he feel the pay is adequate? | ___ | ___ |

Comments: _____

10. Does the operator check sampler performance on a regular basis? ___ ___
    - does he check that hood seals tightly? ___ ___
    - does he check that the sensor grid is warm?
    - what other instrument checks does he perform periodically? _____
    - how often does he check the instrument? _____

| Operator's Duties | Yes | No |
|---|---|---|
| Comments: _____ | | |
| 11. Does the operator maintain a site log book? | ___ | ___ |
| - What type of information is recorded? _____ | | |
| - How often is information recorded? _____ | | |
| Comments: _____ | | |
| 12. Are sample submission sheets correctly and completely filled out? _____ | ___ | ___ |
| 13. Does the Regional Technician visit the site? | ___ | ___ |
| - how often? _____ | | |
| - does he talk to the operator on each visit, or is the sample just left at a location for pickup? _____ | ___ | ___ |

CUMULATIVE PRECIPITATION AUDIT - SANGAMO/MIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| - do the technician and operator discuss sample problems? | ___ | ___ |
| - other than talk to the operator what else does the technician do on his site visits?_____ | | |

Comments: _____

EVENT PRECIPITATION AUDIT - AEROCHEM METRIC

OPERATOR: _____

SITE: _____

| Operator's Duties | Yes | No |
|---|---|---|
| 1. What time does the operator check the sample bag for precipitation? If he cannot change at the proper time, what does he do? (phone technician, change bag when he can, contact alternate operator). _____ _____ _____ _____ | | |
| 2. Does the operator, while removing the sample bag from the sample bucket, allow for any chance of contamination of the precipitation sample? Several points that could lead to contamination are: | | |
| - is the sample bucket removed from the sampler prior to changing sample bag? | ___ | ___ |
| - does the operator wear clean (new) plastic gloves to handle the bag? | ___ | ___ |
| - is the outside of the bag folded up allowing contamination to enter bucket? | ___ | ___ |
| - is the operator careful to not stand directly over sample bucket while removing sample bag? Is there potential for contamination from operator's hair, clothes etc.? | ___ | ___ |

Comments: _____ _____ _____ _____

Concord Scientific Corporation

EVENT PRECIPITATION AUDIT - AEROCHEM METRIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| 3. After the bag is removed from the sample container, | | |
| - is air evacuated from the bag after being removed from container? | ___ | ___ |
| - is the top of the sample bag tied? | ___ | ___ |
| - how far from the top of bag is it tied? | ___ | ___ |
| - what is the sample bag tied with? | ___ | ___ |
| - any chance for other contamination (clothing, etc)? | ___ | ___ |
| Comments: _____ | | |
| 4. For reloading the sample bucket: | | |
| - in placing new bag in sample container, does operator wear gloves? | ___ | ___ |
| - does the operator touch the inside of bag when loading into sample container with only new, clean gloves? | ___ | ___ |
| - any chance for other contamination (clothing, etc.)? | ___ | ___ |
| Comments: _____ | | |
| 5. When the new bag is loaded in the sample bucket: | | |
| - does the bag fit the container well? _____ | ___ | ___ |
| - how much of a decrease in orifice size results from poor bag fit? How much is the sample area reduced (%)? _____ | | |
| Comments: _____ | | |

EVENT PRECIPITATION AUDIT - AEROCHEM METRIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| 6. Describe how the operator transfers the precipitation sample from the sample bag to the sample bottle? | | |
| - does the operator inspect sample bottle for flaws? | ___ | ___ |
| - does he swirl bag and contents around prior to decanting? | ___ | ___ |
| - how is sample melted if partially or completely frozen? _____ _____ | | |
| - does operator pour sample directly from bag into sample bottle? | ___ | ___ |
| - does he use a clean funnel? | ___ | ___ |
| - does he elevate one bottom corner, clean, and cut it? | ___ | ___ |
| - describe cleaning procedure of bag and scissors? _____ _____ _____ | | |
| - if sample volume exceeds bottle contents, is excess poured into graduate and measured? | ___ | ___ |
| - where is the sample transfer area? _____ _____ | | |
| - is a clean sample preparation area used? | ___ | ___ |

Comments: _____
_____
_____

EVENT PRECIPITATION AUDIT - AEROCHEM METRIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| 7. Sample bottles should be labelled to correspond with date submission sheets: | | |
| - what information is placed on sample label? _____ _____ | | |
| - is label placed on cap or bottle? _____ _____ | | |
| - where are samples stored for pickup by technician? _____ _____ | | |
| - how long are samples usually stored prior to pick-up by technician? _____ _____ | | |
| Comments: _____ _____ _____ | | |
| 8. What information is entered on the data sheets? | | |
| - is any information not filled out or incorrectly filled out? _____ _____ _____ | ___ | ___ |
| 9. Is there a log book on site? | ___ | ___ |
| - what information is recorded? _____ _____ | | |
| - how often is information entered into log book? _____ _____ | | |

EVENT PRECIPITATION AUDIT - AEROCHEM METRIC (continued)

| Operator's Duties | Yes | No |
|---|---|---|
| 10. When was the last time technician reviewed sampling procedures with operator? _____ | | |
| - how often are procedures reviewed? _____ | | |
| _____ | | |
| - does the operator think procedures should be reviewed more often? _____ | ___ | ___ |
| - does he think he was adequately trained? | ___ | ___ |
| - does he have any particular problems with the sampling? | ___ | ___ |
| Comments: _____ | | |
| _____ | | |
| 11. Does the operator have sufficient supplies to last for one week, one month, six months? Has the operator ever run out of supplies? _____ | ___ | ___ |
| _____ | | |
| _____ | | |
| 12. What comments does the operator have about the programme? | | |
| - does the operator undertand why he is collecting the sample? _____ | ___ | ___ |
| _____ | | |
| - does he understand why all the special precautions (gloves, cleaning, etc.) are required? | ___ | ___ |
| - does operator get any feedback on programme? Would he like some? How often? _____ | ___ | ___ |
| _____ | | |
| - does he feel the pay is adequate? | ___ | ___ |

CUMULATIVE DRY DEPOSITION - LOW VOL

OPERATOR: _____

SITE: _____

| Operator's Duties | Yes | No |
|---|---|---|
| 1. Describe the procedure used by the operator in handling filter packs. Does he do the following? | | |
| - handle filter packs only by using a plastic bag or on the filter pack? _____ | ___ | ___ |
| - where are the filter packs stored? _____ | | |
| Comments: _____ | | |
| 2. What information is recorded on data sheets? | | |
| - flow rate - start and finish? | ___ | ___ |
| - volumetric reading? | ___ | ___ |
| - time of filter pack changeover? | ___ | ___ |
| - operator comments? | ___ | ___ |
| Comments: _____ | | |
| 3. What training did operator receive on the sampler | | |
| - handling filter packs? _____ | ___ | ___ |
| - has he been retrained since? | ___ | ___ |
| - does he feel training was adequate? | | |
| Comments: _____ | | |

EVENT PRECIPITATION - STANDARD RAIN GAUGE

OPERATOR: _____

SITE: _____

| Operator's Duties | Yes | No |
|---|---|---|
| 1. Is the standard gauge checked each day? | ___ | ___ |
| 2. How does the operator handle the following situation: | | |
| - if water level (meniscus) falls between two graduated marks? _____ | | |
| - what is recorded if level is below 0.2 on scale? _____ | | |
| - if there is water that has overflowed the graduated cylinder, what does the operator do? _____ | | |
| - what is done for freezing rain? _____ | | |

# EVENT PRECIPITATION - NIPHER GAUGE

OPERATOR: _____

SITE: _____

| Operator's Duties | Yes | No |
|---|---|---|
| 1. Does the operator have the following supplied: | | |
| - 2 collection vessels? | ___ | ___ |
| - calibrated volumetric cylinder? | ___ | ___ |
| - funnel? | ___ | ___ |
| Comments: _____ | | |
| _____ | | |
| _____ | | |
| 2. During the time of year when the Nipher gauge is used what does the operator do if it rains? _____ | | |
| _____ | | |
| _____ | | |
| 3. Describe procedure followed if sample volume exceeds graduated cylinder volume? _____ | | |
| _____ | | |
| _____ | | |
| 4. What does the operator do in the case of freezing rain? _____ | | |
| _____ | | |
| _____ | | |

CUMULATIVE WET - TECHNICIAN'S QUESTIONNAIRE

TECHNICIAN: _____

SITE: _____

| Technician's Duties | Yes | No |
|---|---|---|
| 1. How often does the technician visit the site? _____ _____ | | |
| 2. Does the technician park his vehicle upwind of the sampler? Does he leave it running? _____ _____ | ___ ___ | ___ ___ |
| 3. Does he check the seal on the bucket? If there are problems, how does he fix them? _____ _____ _____ | ___ | ___ |
| 4. Does he check that hood delay is 1.5-2 minutes? If the delay is different what steps are taken? _____ _____ _____ | ___ | ___ |
| 5. Does he check inside the instrument housing? (limit switches, clutch, etc.) _____ _____ _____ | ___ | ___ |

CUMULATIVE WET - TECHNICIAN'S QUESTIONNAIRE (Cont'd)

| Technician's Duties | Yes | No |
|---|---|---|
| 6. Does he check the level of all instrumentation? How often does he re-level instrumentation? (Spring only?) _____ | ___ | ___ |
| 7. Does he check the log book to ensure it is up to date and properly filled out? _____ | ___ | ___ |
| 8. Does he discuss the recent sampling period with the operator? How often does he see the operator? _____ | ___ | ___ |
| 9. Are the samples picked up directly from the operator or from a designated pickup location? _____ | ___ | ___ |
| 10. When was the last time the technician reviewed the sampling procedures with the operator? _____ | | |
| Comments: _____ | | |

CUMMULATIVE DRY - TECHNICIAN QUESTIONNAIRE.

| | Yes | No |
|---|---|---|
| 1. How often does the technician visit the site? _____ _____ | | |
| 2. Does the technician park his vehicle upwind of the sampler? Does he leave it running? | ___ | ___ |
| 3. Does the technician check the rotometer setting (2ℓ/min)? | ___ | ___ |
| 4. Does he check for a dark ring on the rotameter flow tube? | | |
| 5. Does he check the tubing seal on the filter pack and on the instrument? | ___ | ___ |
| 6. How often does he calibrate Low-Vol? _____ _____ _____ | ___ | ___ |
| 7. What time of year does he calibrate Low-Vol? _____ _____ | ___ | ___ |
| 8. When was the last time he observed the operator change a filter pack? _____ _____ | ___ | ___ |

# EVENT WET - TECHNICIAN QUESTIONNAIRE

TECHNICIAN: _____

SITE: _____

| Technician's Duties | Yes | No |
|---|---|---|
| 1. How often does the technician visit the site? _____ | | |
| 2. Does the technician park his vehicle upwind of the sampler? Does he leave it running? _____ | ___ | ___ |
| 3. Does the technician check the Aerochem | | |
| Sensor _____ | ___ | ___ |
| Gasket Integrity _____ | ___ | ___ |
| Gasket Cleanliness _____ | ___ | ___ |
| Sample bag fit in container _____ | ___ | ___ |
| Gasket seal _____ | ___ | ___ |
| On each visit? _____ | ___ | ___ |
| Comments: _____ | | |
| 4. How often is the operator checked for operating procedures and retrained? _____ | | |
| 5. How are the samples transported to the laboratory from field site? (boxes, coolers with ice packs, etc.) _____ | | |

EVENT WET - TECHNICIAN QUESTIONNAIRE (CONT'D)

| Technician's Duties | Yes | No |
|---|---|---|
| 6. At the laboratory are the samples weighed? On what type of scale? _____ | ___ | ___ |
| 7. When is the field sample number affixed to the sample bottle? At field site, regional lab or in Toronto? _____ _____ | | |
| 8. How often are supplies brought to the operators, weekly, monthly, yearly? Are they brought out on demand or according to some schedule? _____ _____ | | |
| Comments: _____ _____ | | |

EVENT DRY DEPOSITION - SEQUENTIAL SAMPLER

OPERATOR: _____

SITE: _____

| Technician/Operator's Duties | Yes | No |
|---|---|---|
| 1. Describe operators procedures prior to lowering the boom? Does he do the following? | | |
| - field sheet prepared for each filter pack or tower? | ___ | ___ |
| - are sampling channel flows transferred to data sheet? | ___ | ___ |
| - is the time showing on mechanical clock correct? | ___ | ___ |
| - is the flow rate checked? | ___ | ___ |
| - is the sampler turned off before the boom is lowered? | ___ | ___ |
| Comments: _____ | | |
| 2. After the boom has been lowered, describe the procedure used to reload filter pack. Is the following done: | | |
| - is a new clean sample bag used to remove filter pack? | ___ | ___ |
| - what type of bag is used? _____ | | |
| - are either the filter packs or bags labelled corresponding to sample channel? If not how are the filter packs identified? _____ | ___ | ___ |
| - is the filter pack sampling for that day transferred to another sampling channel? What channel? _____ | ___ | ___ |

| Technician/Operator's Duties | Yes | No |
|---|---|---|
| - is the sampler turned off while filter packs are being removed? | ___ | ___ |

Comments: _____

_____

_____

3. Describe procedure used to reload filter packs. Is the following done:
   - is the filter pack installed on tower while still in bag or is it removed by hand and then installed? _____
   - when are the sampling channels zeroed? _____
   - is the sampler restarted when the boom is on the ground or after being raised? _____
   - does the technician cycle through all channels after sampler is restarted? _____ ___ ___

4. What information is recorded on the sample sheets and in the site log book? _____

5. How frequently is the sampler checked, daily, weekly? Who does the checking, the technician or someone else? _____

EVENT DRY DEPOSITION - SEQUENTIAL SAMPLER (CONT'D)

| Technician/Operator's Duties | Yes | No |
|---|---|---|
| 6. At the laboratory how long do the filter packs usually sit before being unloaded?_____ | | |
| 7. Does the technician unload filter packs in a: | | |
| - laminar flow chamber? | | |
| - fume hood? | ___ | ___ |
| - office? | ___ | ___ |
| - laboratory? | ___ | ___ |
| | ___ | ___ |
| 8. Are there any exposed contaminants in filter processing area? | | |
| - anyone smoking nearby?_____ | | |
| - any exposed chemicals - especially $H_2SO_4$ or $HNO_3$ | ___ | ___ |
| _____ | ___ | ___ |
| - is the area generally clean or dirty?_____ | | |
| Comments: _____ | ___ | ___ |
| _____ | | |
| _____ | | |
| 9. What kind of work surface does the technician use? | | |
| - teflon coated table top? | | |
| - lab bench? | | |
| - lab bench with plastic on top? | ___ | ___ |
| - office desk? | ___ | ___ |
| | ___ | ___ |
| Comments: _____ | ___ | ___ |
| _____ | | |
| _____ | | |

EVENT DRY DEPOSITION - SEQUENTIAL SAMPLER (CONT'D)

| Technican/Operator's Duties | Yes | No |
|---|---|---|
| 10. Is the technician's system for labelling bags easily understandable and logical? (describe the system used) | ___ | ___ |
| - is it done all at once prior to unloading any filter packs? _____ | ___ | ___ |
| - is it done as filter packs are unloaded? _____ | ___ | ___ |
| _____ | | |
| Comments: _____ | | |
| _____ | | |
| _____ | | |
| 11. Describe the procedure used for handling the filters? Are the following points observed? | | |
| - are teflon tweezers used for filter handling? | ___ | ___ |
| - are tweezers stored in methanol prior to use? | ___ | ___ |
| - are they wiped with new Kimwipe before touching filters? | ___ | ___ |
| - are separate tweezers used for impregnated and unimpregnated filters? | ___ | ___ |
| - are Nylon and Impregnated W41 filters folded twice before placement in sample bags? | ___ | ___ |
| - are gloves worn during filter pack unloading? | ___ | ___ |
| Comments: _____ | | |
| _____ | | |
| _____ | | |
| 12. Describe the procedure used for the washing and drying of filters? Is the following done: | | |
| - where are filter samples stored after unloading from filter packs? _____ | | |
| _____ | | |

| Technican/Operator's Duties | Yes | No |
|---|---|---|
| - are filter packs completely disassembled prior to washing? | ___ | ___ |
| - are the filter packs rinsed after washing? What rinses are used?_____ | ___ | ___ |
| - how many of each type of rinse are performed?___ | | |
| - how are the filter packs dried? If air dried, are they covered during drying? What covering is used?_____ | | |
| - if dried in an oven, how long, what oven temperature?_____ | | |
| - are the filter packs used after drying? If not, how are they stored?_____ | ___ | ___ |
| Comments: _____ | | |
| 13. Describe the procedure used for reloading of filter packs. Is the following done: | | |
| - does the technician clean off the area where filter packs will be loaded? | ___ | ___ |

EVENT DRY DEPOSITION - SEQUENTIAL SAMPLER (CONT'D)

| Technican/Operator's Duties | Yes | No |
|---|---|---|
| - is the work surface a lab bench, Teflon overlay board, "Kimwipes" over lab bench or other area? _____ | | |
| - are cleaned tweezers kept in separate breakers - one for unimpregnated and one for impregnated filters? | ___ | ___ |
| - when removing filters from bag does technician push his hand into bag or only tweezers? _____ _____ | | |
| - when placing collar on filter pack is technician careful not to get hand near open exposed face of filter? | ___ | ___ |
| - is the inlet of the filter pack checked to ensure the O-ring has not failed? | ___ | ___ |
| - is the loaded filter pack numbered and stored in a new "Zip-lock" or "Whirl-pak" bag? _____ _____ | ___ | ___ |
| - how are loaded and bagged filter packs stored prior to shipment to field? _____ _____ _____ | | |
| Comments: _____ _____ _____ | | |
| 14. Are any of the filters from Toronto ever damaged? (i.e. nicks out of the sides). If so what is done with them? _____ _____ _____ | ___ | ___ |

DATE DUE

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